FOOD TECHNOLOGY
PROCESSING AND LABORATORY CONTROL

CHEMICAL AND CHEMICAL ENGINEERING SERIES

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CHEMICAL ENGINEERING INSTRUMENTS AND CONTROL METHODS

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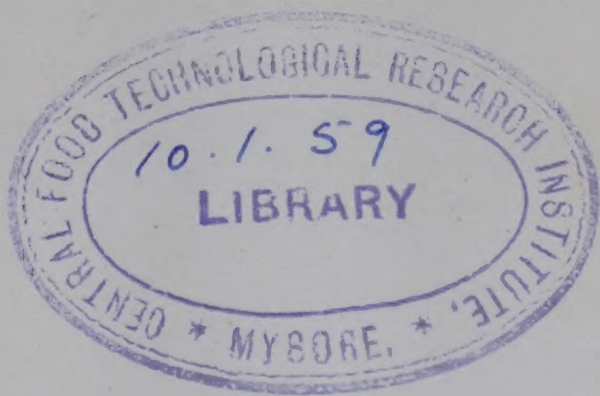## FOOD TECHNOLOGY, PROCESSING AND LABORATORY CONTROL

Advisory Editor: F. AYLWARD, B.Sc., Ph.D., F.R.I.C.

HEAVY CHEMICALS MANUFACTURE AND USES

FOOD TECHNOLOGY
PROCESSING
AND LABORATORY
CONTROL

ADVISORY EDITOR

F. AYLWARD, B.Sc., Ph.D., F.R.I.C.

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PREFACE

FOOD TECHNOLOGY has been defined as "the application of science and engineering to the production, processing, packaging, distribution, preparation and utilisation of foods."

The area of this definition is wide and many text books published are concerned primarily with some one branch—for example with cereal, sugar or meat products. There is, however, an underlying unity in the food industries; based on the nature of the chemical components present in many different foodstuffs, on the engineering techniques employed, on common problems of micro-biology, of hygiene and of nutrition.

In this volume some of the more important food processes have been selected for review and in addition to a description of the techniques used in industry, details have been given of analytical and other control methods. The authors all have practical experience in their special fields and have described the analytical methods adopted by their own laboratories.

It is hoped, therefore, that this volume will be of use to chemists engaged in food processing, as well as to students preparing for careers in the food industries.

FRANCIS AYLWARD.

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PART 1.—PROCESSING METHODS

CHAPTER 1

SUGAR REFINING

Raw sugar used for refining in the United Kingdom is derived from sugar cane and sugar beet.

Raw cane sugar is imported from tropical or sub-tropical countries, principally from Cuba, British West Indies, British Guiana, Australia, Natal, Mauritius, Fiji and San Domingo.

Raw beet sugar in pre-war days was purchased from the Continent of Europe but at the present time it is largely produced in the United Kingdom. The appearance of both types of raw sugar is not very different. The colour is light yellow to brown and the size of the crystals is variable. More or less raw syrup or molasses still clings to the surface of the crystals, to which the colour and stickiness are due.

Raw sugar cane smells and tastes quite pleasant, but raw beet sugar, especially when of low purity, is decidedly unpleasant.

The Sugar Cane

The juice in the sugar cane is expressed in roller mills. The cold juice is heated and lime is added, which combines with some of the impurities and coagulates them. After settling, the clear juice is run off. Alternatively, if the juice is limed cold and then heated, the dissolved air is expelled and rises, carrying the impurities to the top as a scum, which is removed, leaving clear juice. The juice is concentrated in evaporators, boiled to form crystals in steam-heated vacuum pans, and the mixture of crystals and mother syrup is spun in centrifugals to separate the raw cane sugar from the cane molasses.

The Sugar Beet Roots

Sugar beet roots that have been topped and tailed in the fields are washed free from earth and stones, sliced into thin chips (cossettes) which are fed into large vessels termed diffusers. Hot

water is passed through them causing the sugar contained to diffuse into the water, leaving a large portion of the soluble but non-diffusable impurities behind. The sweet water obtained passes through a series of these diffusers "a battery," becoming increasingly richer in sugar until finally its concentration nearly reaches the concentration of the sugar in the juice of the original root. Various types of continuous diffusers are gradually superseding the older battery system in Europe. Although constructional details vary, the principle is similar, i.e., of cossettes and juice travelling in opposite directions in a sloping cylindrical vessel.

This sugar solution or so-called "juice," is then treated with lime and gaseous carbon dioxide or gaseous sulphur dioxide according to whether the carbonatation or sulphitation process is utilised. A precipitate of carbonate or sulphite of lime is produced which removes some impurities from solution. This precipitate is separated from the clear light-coloured juice by filter presses and the juice is then concentrated in evaporators.

The thick juice is treated in the same way as thick cane juice, but yields in this case raw beet sugar and beet molasses.

THE PURPOSE OF THE REFINING PROCESS

The refining process removes reducing sugars, ash and other organic matter almost completely, thus giving refined white sugar, which is sucrose of a purity above 99.9 % on dry matter.

As so much misconception prevails regarding the various forms of sugar, it is only fitting to describe briefly the objects of refining.

The Raw Sugar

Raw sugar or "brown unrefined sugar" is impure and may contain sugar lice, "Acari," mould spores which cause deterioration of cooked foods, bacteria which produce acidity in milk and the like, and dirt and clay, pieces of wood, string, particles of cane fibre, and sometimes even large insects, beetles and small reptiles.

Raw sugar becomes moist if the atmosphere is moist, as the surrounding film of molasses is hygroscopic. It becomes then an ideal breeding ground for micro-organisms. It is therefore an impure sugar.

TABLE I
ANALYSIS OF RAW SUGARS

	Cuban	Javan	Australian	West Indian	British Beet
Sucrose – –	96·70	97·10	98·60	96·50	97·09
Reducing sugars –	0·92	1·24	0·38	1·45	0·04
Ash – – –	0·52	0·35	0·23	0·41	0·54
Organic non-sugar	1·13	0·70	0·48	0·54	1·06
Water – – –	0·73	0·61	0·31	1·10	1·27
	100·00	100·00	100·00	100·00	100·00

TABLE II
ANALYSES OF RAW AND AFFINED CANE SUGARS (DRY SAMPLES)

	Raw	Affined
Sucrose . . .	97·54	99·44
Reducing sugars .	·74	·16
Ash. . . .	·68	·10
Organic non-sugar .	1·04	·30
	100·00	100·00

The Refined White Sugar

Refined white sugar is an article of food which is free from impurity to an extent unapproachable by any other foodstuff made in such large quantities. Sugar is not refined solely for colour but to make it a clean, dry, sound food, of exceptional chemical and bacteriological purity. Refining does not deprive sugar of its sweetness or nutritive value. Pure sucrose can only exist in the form of hard, dry, white crystals and in such form it is of maximum sweetness. Whether the refined sugar has been derived from raw beet sugar or raw cane sugar makes no difference. It is impossible to determine the origin from the analysis or sweetness of the refined white sugar.

Cube Sugar

Cube sugar is no sweeter nor less sweet than refined white granulated sugar. It has been produced from the same liquor by boiling, and the sole essential difference in manufacture lies in the centrifuging of the massecuite in special machines which produce

thin slabs of moist sugar which, after stoving to dry, are chopped into the familiar cubes. Its higher price is due to this more elaborate manufacture.

STAGES IN THE REFINING PROCESS

Until recently all raw sugar arrived at the refinery in jute bags holding 1 to 3 cwts. For reasons of economy this is being displaced by bulk loading of ships and bulk unloading by power grabs which discharge on to conveyors which deliver the raw sugar to large shed stores or to silos, or directly to the refinery.

The First Process—Mixing With Raw Syrup

The raw sugar is conveyed by travelling bands either direct or from silos to the affination house, where it is mingled with raw syrup to form a fluid "magma" of crystals and syrup. This is passed by means of screw conveyors to centrifugal machines rotating at high speed, where the syrup is thrown off through the perforated metal sides, leaving a wall of sugar which is washed with a jet of hot water to remove adherent syrup.

This affined sugar is of lighter colour, as most of the impurities have been removed.

The Syrup and Washings Are Now Boiled to Grain

Some of the syrup and washings is circulated for making magma, but the excess is boiled to grain in vacuum pans. The resulting mixture of crystals and mother syrup, "massecuite," is centrifuged like raw sugar, and joins the affined sugar in the melter. The mother syrup is boiled again to yield a further crop of recovered sugar and final syrup or molasses. This molasses is a syrup of such a very high non-sugar content that it is uneconomical or impossible to remove further sucrose by boiling.

Removing the Coarse Insoluble Impurities

The affined sugar and recovered sugars are dissolved in hot water in a tank "melter" and passed through a wire mesh strainer to remove coarse insoluble impurities.

Removal of Further Impurities

The strained melted liquor is further purified by one or other of the following methods: Kieselguhr filtration without previous

FIG. 1.—AFFINATION
MAGMA OF RAW
SUGAR AND RAW
SYRUP CHARGED INTO
A HIGH-SPEED
CENTRIFUGAL
MACHINE.
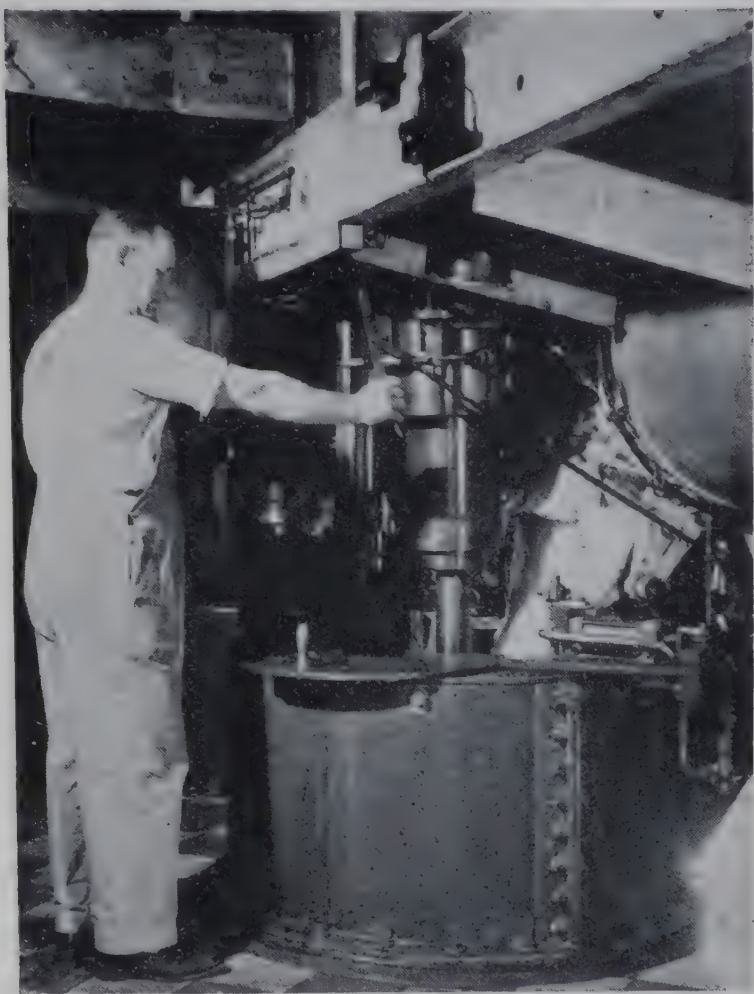
treatment or with phosphate defecation. Both methods are common in the United States. The Williamson process of forming a floc of precipitated phosphate of lime which is floated off as a blanket of froth caused by aeration is used in Louisiana and is gaining favour elsewhere. Most British refineries and some Canadian and Australian utilise the carbonatation process. The earliest form of this consisted of treating melter liquor in batch carbonatation tanks with lime cream and carbon dioxide gas whilst heating to 90° C. The precipitate of carbonate of lime thrown down adsorbed colouring matter and other soluble non-sugar impurities and also served as a filter aid in pressure filters, yielding a lighter colour liquor, "carbonated liquor" or "brown liquor" entirely free from insoluble impurities.

A development of the batch carbonatation method is that of continuous carbonatation in which liquor, lime cream and carbon dioxide are led simultaneously into a tall tank and the overflow further treated with carbon dioxide in successive tanks. By

this means a coarser precipitate easier to filter is obtained. The carbonated filtered liquor is then ready for decolorisation.

The Bone Charcoal Treatment for Decolorisation

This is effected by treatment with bone charcoal. The preparation of bone charcoal for sugar decolorising is not carried out in a refinery, but is an entirely separate undertaking. Carefully selected bones are degreased, broken to correct sizes and heated in closed retorts to carbonise the organic constituents of the bone, leaving about 10 per cent carbon on a porous inorganic framework chiefly composed of tri-calcium-phosphate. This material is capable of adsorbing colouring matters and other organic and inorganic non-sugars from sugar liquors.

Tall vertical cylindrical cisterns containing 30 to 40 tons of bone charcoal are filled from brown liquor tanks through a convenient arrangement of pipes and valves termed an "organ." Brown liquor is slowly run through the cisterns and emerges from a narrow pipe at the bottom of each cistern into a shallow tank called a "rhone." The emergent liquor known as "fine liquor" is water white at first, but after the cistern has been running some time, the decolorising value of the charcoal decreases and tinted liquor is obtained. Finally the liquor is too coloured for use, so that brown liquor has to be shut off at the organ and hot water turned on to displace the liquor and ultimately wash off all traces of sugar.

Water washing is continued until adsorbed impurities are no longer removed.

The Wet Char is Next Removed to the Revivification Kilns

The water supply is then cut off at the organ, the cistern drained and the wet char allowed to run from a manhole at the base on to a belt conveyor, which carries it to the top hopper of revivification kilns. These consist of a number of long vertical pipes passing through a fire of regulated intensity. The pipes stretch through the fire bed to some distance below, to cool the char before it tumbles into the revivified bin.

Newer design of kilns are displacing the old pipe kilns. The Herreshoff multi-bed roasting kiln originally devised for ore treatment and the Stordy kiln, with more uniform heat distribution specially designed for bone charcoal, are both claimed to

FIG. 2.—BROWN LIQUOR TANKS FEEDING BONE CHARCOAL CISTERNS.

give more uniformly regenerated charcoal with possibly less mechanical breakdown of the particles.

The Revivification Kilns

The function of the kilns is firstly to dry the char, and secondly to burn off adsorbed organic colouring, etc., which has not been removed by the wash water. The revivified char, after passing over sieves to remove fine char dust, is refilled into cisterns for further decolorisation of liquor. The char dust which has been removed is a useful by-product and is used as a source of phosphate of lime for mineral feeding of animals, for fertilising the soil, or as a pigment.

How Refined Sugar is Obtained from the Fine Liquor

Returning to the fine liquor from the char cisterns, we will follow its course towards its end, namely refined white sugar.

It is pumped from the rhone to tanks on the pan floor and there drawn into steam-heated vacuum pans, where it is boiled at low temperature to evaporate water without damaging or colouring the liquor.

The Formation of the Sugar Crystals

Whereas this operation was formerly due solely to the skill of the pansman in operating his valves, it is now generally achieved
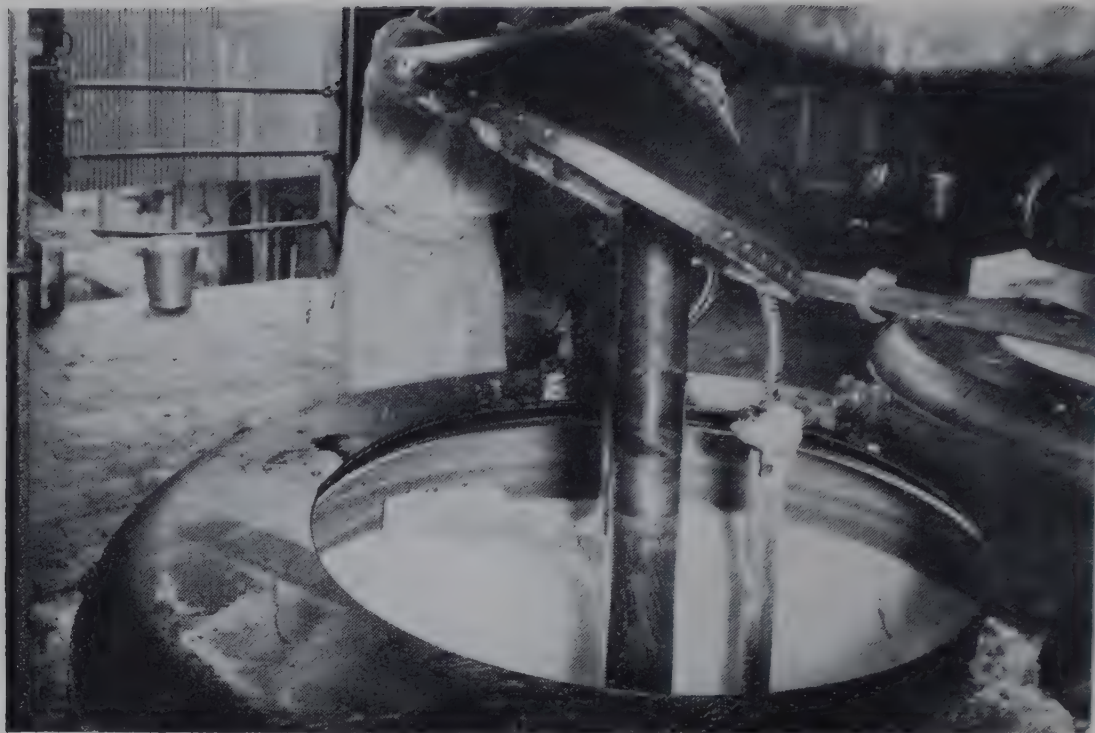
FIG. 3.—DISCHARGING SUGAR FROM CENTRIFUGAL MACHINE.

by instruments which enable him to control the degree of super-saturation within the limits suitable for crystal growth and also to inspect the size of the crystals whilst boiling is in progress.

When the pan contains its maximum working capacity load of "massecuite," i.e., crystals and mother syrup, the steam is shut off, the vacuum broken and the contents of the pan discharged through troughs into large tanks containing stirring gear, which keeps the massecuite in movement until it is drawn into centrifugal machines.

Here the mother syrup is spun off through the perforated wall of the centrifugal basket; a jet of boiling water is used to wash the face of the wall of white sugar left lining the basket, and when these washings have been spun off, the machine is stopped and the sugar ploughed out through the circular opening at the bottom, which previously is kept closed by a bell-shaped cover.

The Rotating Dryers

The hot and moist white sugar falls into a conveying trough, which quickly carries it to the granulators, which are long cylindrical rotating dryers arranged at a slight angle to the horizontal. The sugar is fed into the raised end, a series of vanes in their rotation lift the sugar to the top position and allow it to

fall continuously in a cascade, whilst heated air is passing through the granulator, carrying away moisture with it. This operation continues until finally the sugar arrives at the lower end and falls as dry crystal granulated sugar on to a belt conveyor which carries it on to grading sieves and thence to the filling house.

Before consideration of the packing, a return will be made to the mother syrup and washings from the refined sugar centrifugals.

Treatment to Produce Further Yields of Sugar

As this mixture of mother syrup and washings contains 40 to 50 per cent of the sugar originally contained in the fine liquor from which it was boiled, a further boiling is necessary to recover another crop of white sugar. The syrup and washings from this second crop are decolorised in char cisterns to remove some of the colour and non-sugars that have become concentrated in these mother syrups, together with the small amount of colour that has been produced in boiling in vacuum pans.

The decolorised material is then cropped again and again yielding further white sugar until finally the sugar obtained is no longer white and is remelted and once more put through the refining process or is sold as refinery soft sugar or pieces.

Packing the Sugars

The combined yields of white sugars, granulated and graded, arrive at the fill houses, and a portion diverges to the bulk filling, where it is automatically filled and weighed into 1 or 2 cwt. bags and machine sewn. A modern development for the special benefit of food manufacturers is the delivery of loose sugar in road tanks. The remainder is conveyed by travelling belts to a series of hoppers, arranged above ingenious machines, which convert rolls of thin blue cardboard into open cartons labelled ready for filling, on one section, and are quickly and automatically transferred to the other section in which 1, 2 or 4 lb. of sugar are automatically weighed and delivered into the cartons, which the machines then close and seal and pass to tables where they are parcelled ready for supply to grocers and thus to the consumer.

At no stage, from the pan boiling to final carton, is the refined sugar touched by the hand, so that its final cleanliness and purity is assured.

H. C. S. de W.
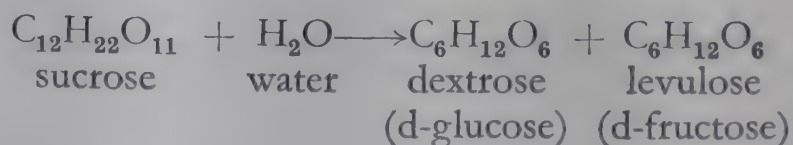
B

SUGAR CONFECTIONERY

In this chapter no attempt will be made to cover all the many aspects of sugar confectionery, but examples will be given to illustrate methods of manufacture.

HIGH BOILINGS

This section will be treated more fully than the others, not only because boiled sweets form the major part of confectionery production, but because the behaviour of the sugars is common to many other products which follow later.

Hard boilings may be made by boiling sugar and water to such a temperature that practically no water remains—on cooling, the mass remains in a vitreous state. The result is actually a very supersaturated solution of sugar, since at room temperature a saturated solution of sucrose contains only about 66·5 per cent of sugar. Ordinary granulated sugar (sucrose) is not capable of being made into boilings by itself—it crystallizes out of solution, or "grains", far too easily.

However, sucrose may be converted into other sugars by a process of hydrolysis—usually called the "inversion" of sugar:

$$C_{12}H_{22}O_{11} + H_2O \longrightarrow C_6H_{12}O_6 + C_6H_{12}O_6$$

$$\text{sucrose} \qquad \text{water} \qquad \text{dextrose} \qquad \text{levulose}$$
$$\text{(d-glucose)} \quad \text{(d-fructose)}$$

These two sugars, dextrose and levulose, are produced in equal amounts and are together called "invert sugar."

Invert sugar has the property of stabilising the supersaturated solution of sucrose and so reducing the tendency to grain. Sufficient invert sugar must be produced during manufacture, but this must be carefully controlled because the levulose in invert sugar is very hygroscopic: too little invert sugar and the sweet will grain, too much and it will pick up moisture very quickly. 10 to 15 per cent of invert sugar has been found to be

the optimum figure. Here is a case where laboratory control is necessary to achieve uniformity.

The original method of making boilings was to add sucrose to water and to boil the resulting syrup on coke or gas stoves until less than 2 per cent water remained (300° F. to 330° F.). In order to catalyse the inversion of the sucrose an acid must be added—usually a very weak one, such as cream of tartar. The exact amount depends on many factors—in particular the hardness of the water used and the exact time at which it is added to the syrup during the boiling period—the weight can only be determined by trial and error but is of the order of 0·15 to 0·25 per cent (in terms of sugar). Once a given procedure has shown that the correct amount of inversion takes place (about 10 to 15 per cent), that procedure must be rigidly adhered to—even to the extent of using the exact amount of water each time. Often more than half the cream of tartar is used in neutralising the hardness of the water.

It is unfortunate that it is not possible to calculate the amount of invert sugar which should be produced under given conditions. Undoubtedly the hydrogen ion concentration (pH) is one of the most important considerations but information is lacking on the pH of acids at high temperatures and in the presence of high concentrations of sugar—particularly as temperature and concentration are increasing all the time. It is essential therefore to institute regular checks on the invert content.

From what has been said above the obvious question may seem to be—why not add a measured amount of invert sugar, previously prepared, to the batch before cooking? It is not easy to give an answer to this but two points may be made:

(a) A certain amount of invert sugar is produced from sucrose by the action of heat and the mineral matter present. This will occur in any case and so not much improvement in standardisation will occur.

(b) The levulose in invert sugar is susceptible to heat and over a period of time will break down. This results in an appreciable deepening of the colour.

Moreover, the laevulic acid produced is able to invert more sucrose and this effect will be magnified if the invert sugar is added all at once at the beginning of the boil, instead of being produced continuously as boiling proceeds.

Starch Syrup

Fortunately another material has been found to replace invert sugar. This is a syrup produced by the hydrolysis of starch (using enzymes or acids) and consists of the sugars dextrose and maltose together with dextrins.

This product is known as *starch syrup* or *corn syrup* or *glucose syrup* and is often referred to as *confectioners' glucose*. In view of the fact that the term glucose refers to a definite chemical compound, it seems preferable to use the name *starch syrup* for the mixture obtained by hydrolysis of starch.

By careful control during manufacture the syrup is supplied as a standardised product, and a typical analysis is:

dextrose   16 per cent
maltose   27 per cent
dextrin   40 per cent
water   17 per cent

By adding starch syrup to sucrose in the boil (about 30 to 40 per cent) graining is hindered. As the syrup does not contain the hygroscopic and heat labile levulose, it can be added in a measured amount to produce a standard product. Three
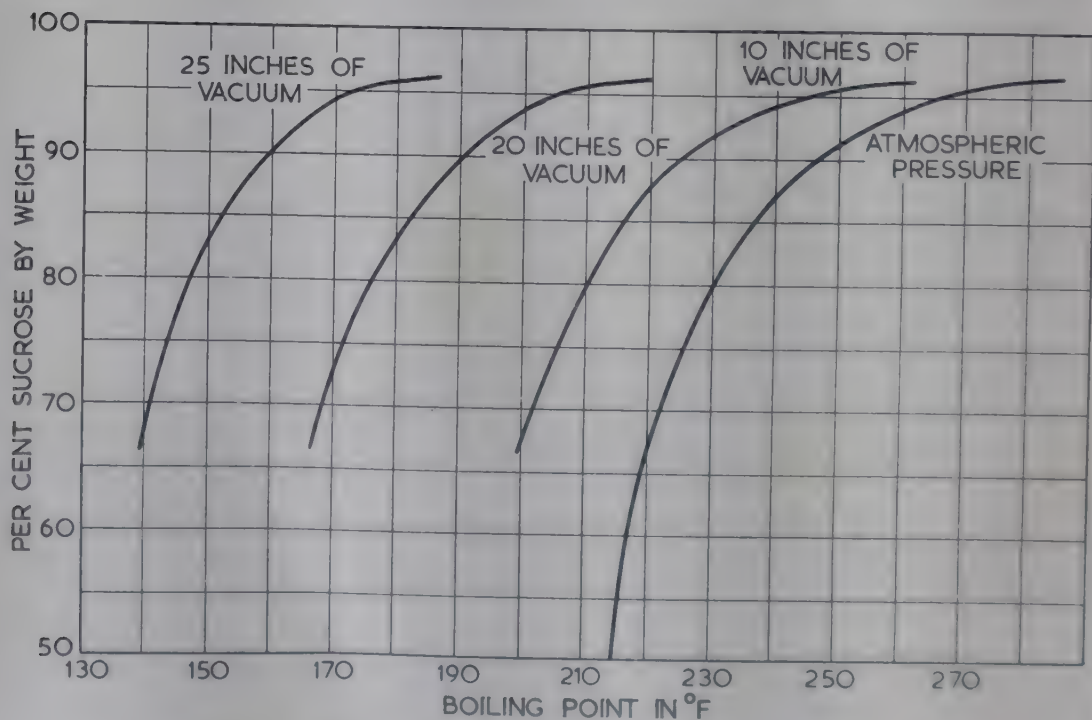

FIG. 1.—BOILING POINTS OF SUCROSE SOLUTIONS.

differences in its effects may be noted: (1) It is less sweet than sucrose and invert sugar; (2) The dextrin present imparts a toughness to the sweet; (3) Inferior starch syrup sometimes gives a slight cloudiness on boiling. For these reasons it is generally held that sucrose-invert sweets (commonly called "all sugar" boils) are superior to those from sucrose-starch syrup.

Methods of Cooking

When cooking is carried out on gas stoves, the batch sizes are limited to about 30 to 40 lb., and thus the rate of production is low. Although the best work is still done in this way, cooking by steam has now become the usual process.

Unfortunately an open steam pan is impracticable as, with the highest steam pressure usually available (120 p.s.i.), the highest temperature which can be reached is in the region of 280° F. to 290° F. depending on the pan, and this may take 30 to 60 minutes.

An answer was found, however, in cooking under reduced pressure (Fig. 1). There are two types of vacuum cooker—batch and continuous. In the *batch type* the syrup is boiled to about 260° F. to 270° F. under atmospheric pressure and is then subjected to reduced pressure for a given time. The exact details depend on the type of machine. In the *continuous type* (Fig. 2) the syrup is pre-cooked in open pans to 230° F. to 240° F. and is then pumped through a steam heated coil where it is cooked to about 240° F. under a vacuum equivalent to 25 to 28 inches of mercury and is withdrawn continuously. These cookers are capable of producing up to 1 ton of boiled sugar per hour. Their advantage is that colour and invert sugar production are kept to a minimum, the latter being less than 2 per cent. A disadvantage is that the sugar mass at a temperature of only 240° F. is much more viscous and tends to retain air bubbles more than fire boils.

Starch syrup is almost always used in vacuum work—with lower temperatures and very short cooking times much larger amounts of inverting agents would be required and control would be even more difficult.

Cooling and Manipulation

The cooked sugar, either gas or vacuum boiled, is pitched on to an oiled and heated slab, and colour, flavour and acid are
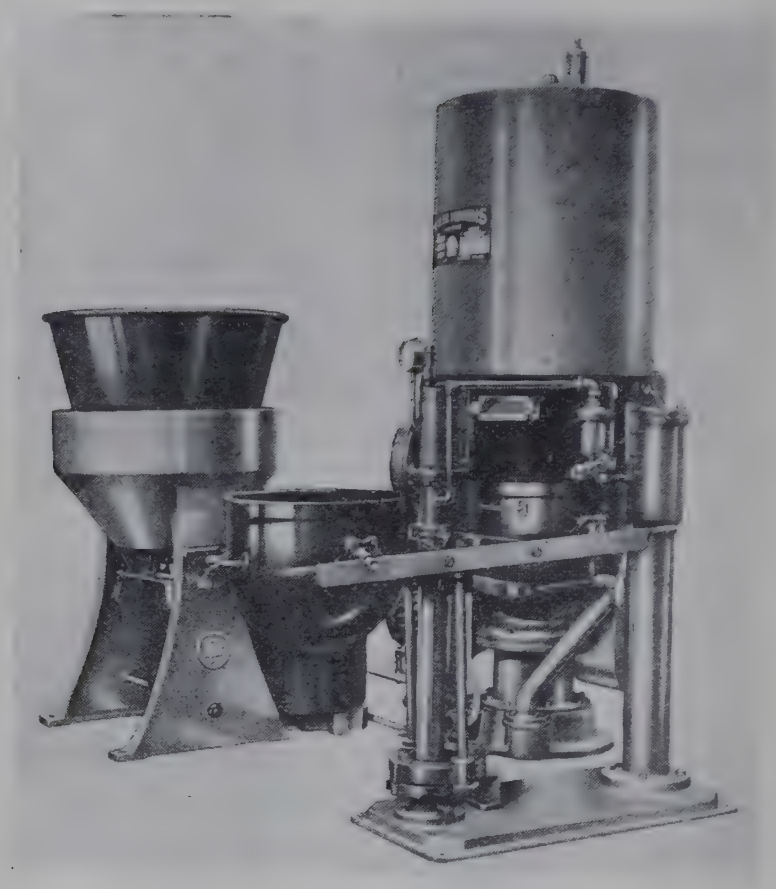
Fig. 2.—Continuous Vacuum Sugar Cooker.
(Baker Perkins, Ltd.

mixed in. Manipulation is best kept to a minimum since handling increases the number of air bubbles occluded and also tends to induce graining if the proportion of glucose or invert is low. Although colour may be added to the batch before cooking, acids and flavours cannot be added in this way.

No satisfactory method of incorporating acids and flavours, except by kneading on the slab, has been found.

Colour.—This should preferably be in liquid form, dissolved in distilled water, the solubilities of different colours being carefully taken into account. If this is not possible a paste should be made with colour, water, glycerine and glucose.

Flavours.—These should be of the type specially made for boilings: if not, volatility may be reduced by mixing with triacetin or a similar substance.

Acid.—This is usually added dry. It should be of small crystal size and free from lumps.

When the batch has cooled somewhat, usually to about 180° F., it is ready to be formed into individual sweets. For *drops* it is merely passed through rollers bearing the appropriate
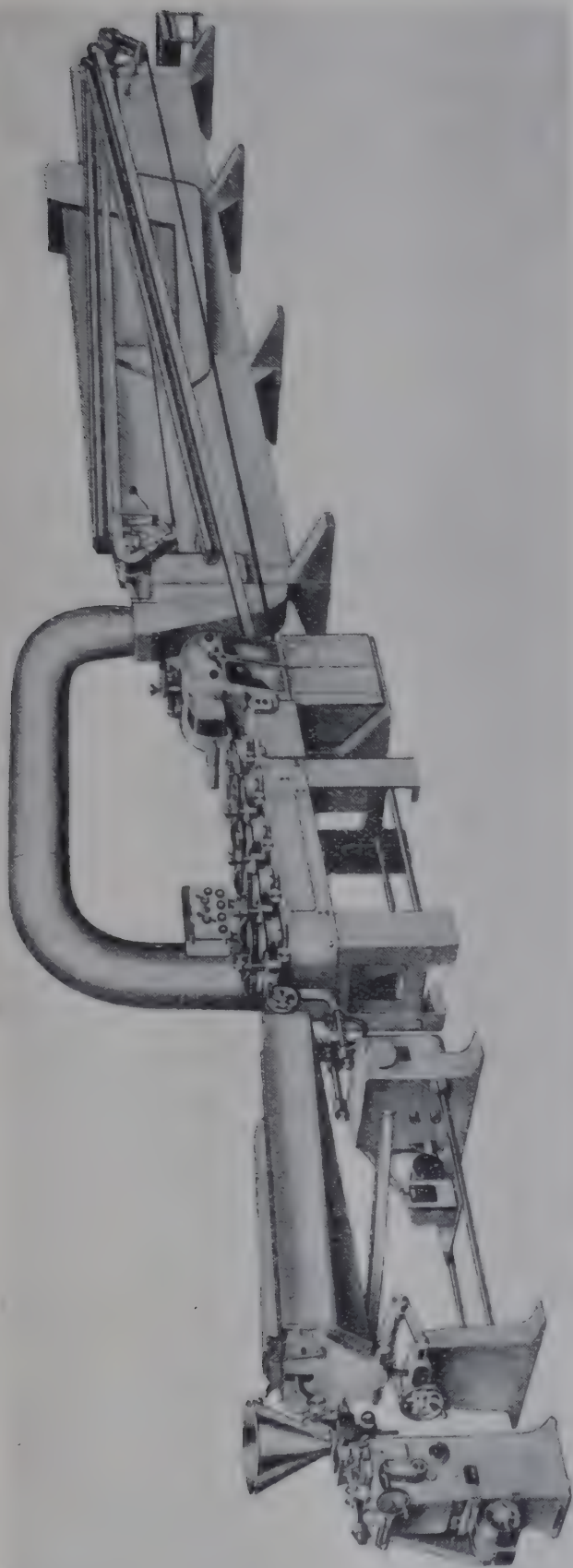
Fig. 3.—Centre Filler, Batch Roller, Rope Sizer, Forplast Forming Machine and Multi-Band Cooler in Series.

The centre filler continuously pumps the centre filling into the rope as it is formed. After forming, the sweets are cooled by means of a multi-band cooler down to a temperature of 100–110° F.

(Baker Perkins, Ltd.)

die. Flour is often used for dusting purposes but its use should be kept to a minimum as it causes a roughness in the sweets.

For *centre* sweets the mass is placed in a batch roller where it is spun out into a rope, passed through sizing rollers and thence to the die. A variety of centres may be incorporated on the slab, or if sufficiently fluid by means of a centre filler. This machine continuously pumps centre into the rope as it is formed (Fig.3). The centre should be at approximately the same temperature as the sugar mass otherwise cooling and cracking will occur before the sweet is formed. If the centre contains water (e.g., as in jam) the moisture content should not exceed 12 per cent or graining will be induced inside the sweet.

After forming, the sweets are cooled by means of an air blast. This should be such that the sweets are cooled to about 100° F. to 110° F. before discharge. Above this temperature they may stick together and below it they may pick up moisture. On cooling, the thin "web" of sugar, which joins the sweets together when they are made, breaks and falls away. Sweets should preferably be packed or wrapped while slightly warm—at about 90° F. to 100° F.

FONDANT

One of the basic processes in confectionery manufacture is the making of fondant. Fondant consists of a saturated sugar syrup containing a very large number of minute sucrose crystals. It may be prepared by boiling sugar and water, to about 245° F., cooling quickly to 100° F. to 110° F. (to produce a supersaturated solution) and then agitating violently. The agitation induces the excess sucrose to crystallise in very fine crystals: it also incorporates air which makes the product appear more white.

In practice starch syrup and/or invert sugar is added before cooking since fondant made with sucrose alone has a hard texture and dries out rapidly. There is also the possibility of fermentation, which occurs in the syrup phase. It has been shown that a concentration of 76 to 78 per cent solids in a syrup is necessary if fermentation is to be completely prevented, in the absence of acids. With fondant made from sucrose alone the solids in the syrup phase are only about 66·5 per cent. Addition of starch syrup or invert sugar raises the concentration of sugars and lessens the risk of fermentation. From this point of view
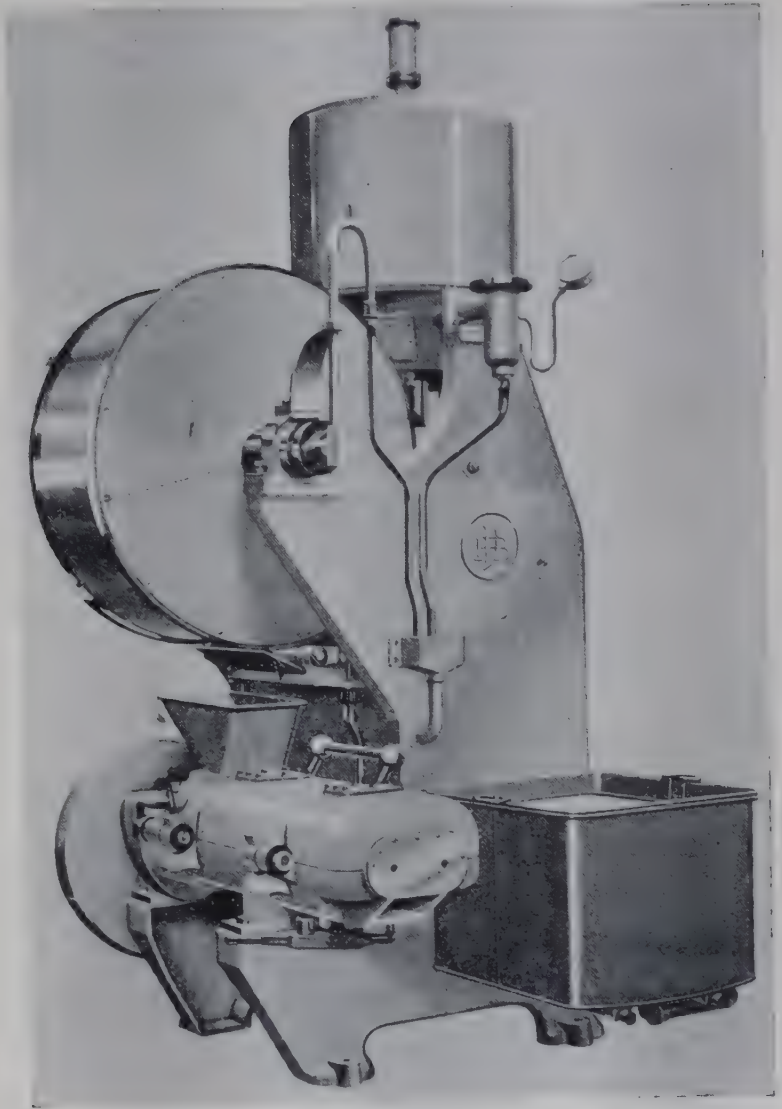
FIG. 4.—
CONTINUOUS FONDANT
MAKING PLANT.
(Baker Perkins, Ltd.)

fondants containing more than six parts of sucrose to one of starch syrup are not recommended.

The most general method of fondant manufacture is by means of the continuous fondant cooker (Fig. 4). Sugar, starch syrup and water (preferably softened) is prepared in the dissolving pan and brought to boiling point, taking care that all sugar is dissolved. The syrup is pumped to a continuous cooker where it is boiled to 242° F. to 248° F., according to requirements, under atmospheric pressure. The cooked syrup falls on to a rotating water-cooled drum which quickly cools the syrup in a thin sheet without crystallization. The cooled syrup is scraped off and falls into the beater where crystallization and aeration occur. Much heat is evolved, due to friction and release of latent heat. For the best results close attention should be paid to temperature.

The syrup should enter the beater at 100° F. or less and the emerging fondant should not be higher than 120° F.

FONDANT CREAMS

One of the main uses for fondant is the production of "cream" centres. The simplest method is to re-heat the fondant, add colour and flavour and pour into suitable moulds. The temperature should not exceed 150° F.—except where the creams are to be wet crystallized when temperatures up to 180° F. are used—as the temperature is increased more and more sugar dissolves and on cooling it tends to crystallize again in large crystals which spoil the texture.

The fondant is usually too viscous to pour properly by itself and is "thinned" by the addition of sucrose or invert syrup. On no account should *water* be used for the dilution, since water will dissolve additional crystalline sugar, increasing the syrup phase and lowering the concentration of solids in it. The cream will not set properly and will be more open to fermentation.

If acid is added to the batch, it is best to use a 50 per cent solution. Occasionally frappé is added to the batch, just before pouring, to lighten it. Frappé is a mixture of egg white and sugar syrup whipped till its density is of the order of 5 pounds per gallon (due to aeration).

Deposition into Moulds

When prepared, the fondant is deposited into rubber or starch moulds. The latter method lends itself to large scale production by means of a *mogul machine*. This machine is fed with trays of previous work. It empties the trays and brushes the goods free from starch. The empty trays are re-filled with starch, levelled and printed with the required shapes, which may be up to 180 per tray. The trays pass under a depositor which drops a carefully adjusted amount of cream into each mould. The trays are returned to the machine when the cream has set. The moisture content of the starch should be between 5 and 8 per cent.

Wet Crystallization

The creams are either chocolate covered or wet-crystallized. The latter method briefly is to prepare a supersaturated solution

of sucrose which is poured over the creams. As the sucrose crystallizes out it deposits a layer of crystals over the surface of the sweet. The sugar is dissolved in water to 71 to 74 per cent concentration by heating, depending on the time for which it will be left on the creams. It may be cooled with stirring down to just above its saturation temperature (Fig. 5) but below this all agitation must be avoided to prevent premature crystallization.

JELLIES AND GUMS

These types of confectionery may also conveniently be deposited into starch. However, the starch must be warm (about 120° F.) and its moisture content must be controlled (to 4 to 5 per cent) to produce clean work.

Soft Jellies

Agar-agar and *pectin* are used to produce soft jellies. The agar must be soaked before use for at least twelve hours in 40 to 50 times its weight of water. The water is drained off, fresh water added and heated to dissolve the agar. Sugar and starch syrup are added and the batch is boiled to 226° F. and colour and flavour are then added. Prolonged heating destroys the gelling power of agar so that the batch must be cooled (preferably in a
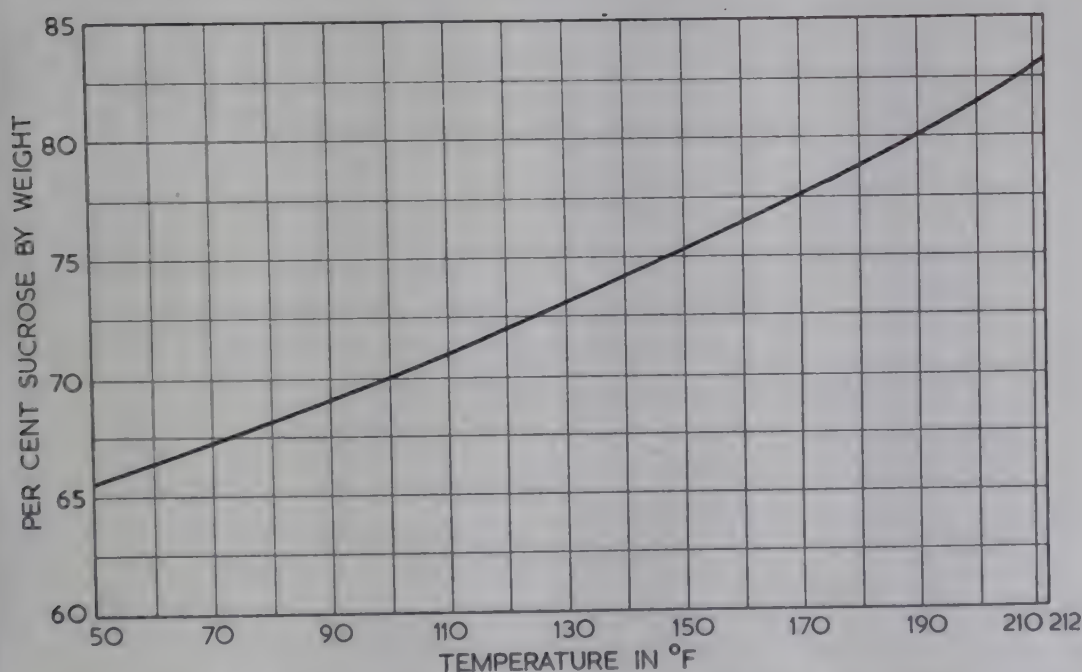
FIG. 5.—SATURATION TEMPERATURES OF SUCROSE SOLUTIONS.

water-jacketed pan) to 140° F. before depositing. To minimise hydrolysis the acid is added just before depositing in starch.

Pectin is normally available in powder form. It is dispersed dry in part of the granulated sugar and the mixture added to vigorously boiling water. The batch is then prepared as for agar, except that cooling to 140° F. is unnecessary. Fruit pulps or jams are sometimes added to jellies: the method of addition should be carefully studied as their acidity may produce over-inversion of sucrose in the batch, leading to sweating.

After standing in starch the jellies should have a moisture content of 20 to 22 per cent. This can be checked on a projection refractometer (see p. 174): indeed, this instrument is the best means of controlling the cooking of the jellies. After brushing off, the jellies are either chocolate covered or dry crystallized— this latter merely involves moistening the surface with a steam jet, rolling in granulated sugar, re-steaming and drying. It is much quicker and cheaper than wet-crystallizing but is less effective.

Hard Gums

These are produced with either gum arabic or gelatin. The gum is dissolved in water and strained to remove foreign matter. The gelatin is soaked in water. A syrup is prepared by mixing sugar and starch syrup and boiling to about 250° F.; gum or gelatin is added, followed by colour and flavour. The batch is cast into starch and the trays are placed in a stove at 120° F. for 3 to 10 days until the required texture is obtained.

TOFFEE, CARAMEL AND FUDGE

These differ from the products so far described in that, besides sugar and starch syrup, fat and milk are also employed. There is no hard and fast distinction between toffees and caramels, although generally toffees are boiled slightly higher—260° F. to 270° F. against 250° F. to 255° F. for caramels. The usual method of testing is to take a small piece of the boiling batch and to cool it under water, when the texture is judged—"soft ball"— "hard ball"—"soft crack"—"hard crack". This is reasonably satisfactory in experienced hands and with water at a constant temperature, but the use of a suitably installed thermocouple thermometer has much to commend it.

The toffees or caramels are poured on to an oiled slab, cooled, passed between sizing rollers and cut into pieces: or alternatively fed into a "cut and wrap" machine (Fig. 6) in which the mass is extruded in the form of a rope, cut into pieces and wrapped, at rates of up to 650 pieces per minute.

The following points are worthy of note:

A. SUGAR.—In order that the batch shall not grain the sugar must be properly dissolved. This is not easy if the recipe states that all ingredients shall be mixed warm at first, to aid fat emulsification, without adding water. If the mixture is brought quickly to the boil there is a danger that some sucrose may remain undissolved.

B. STARCH SYRUP.—This is usually about one third of the batch, although the proportion may be increased for "cut
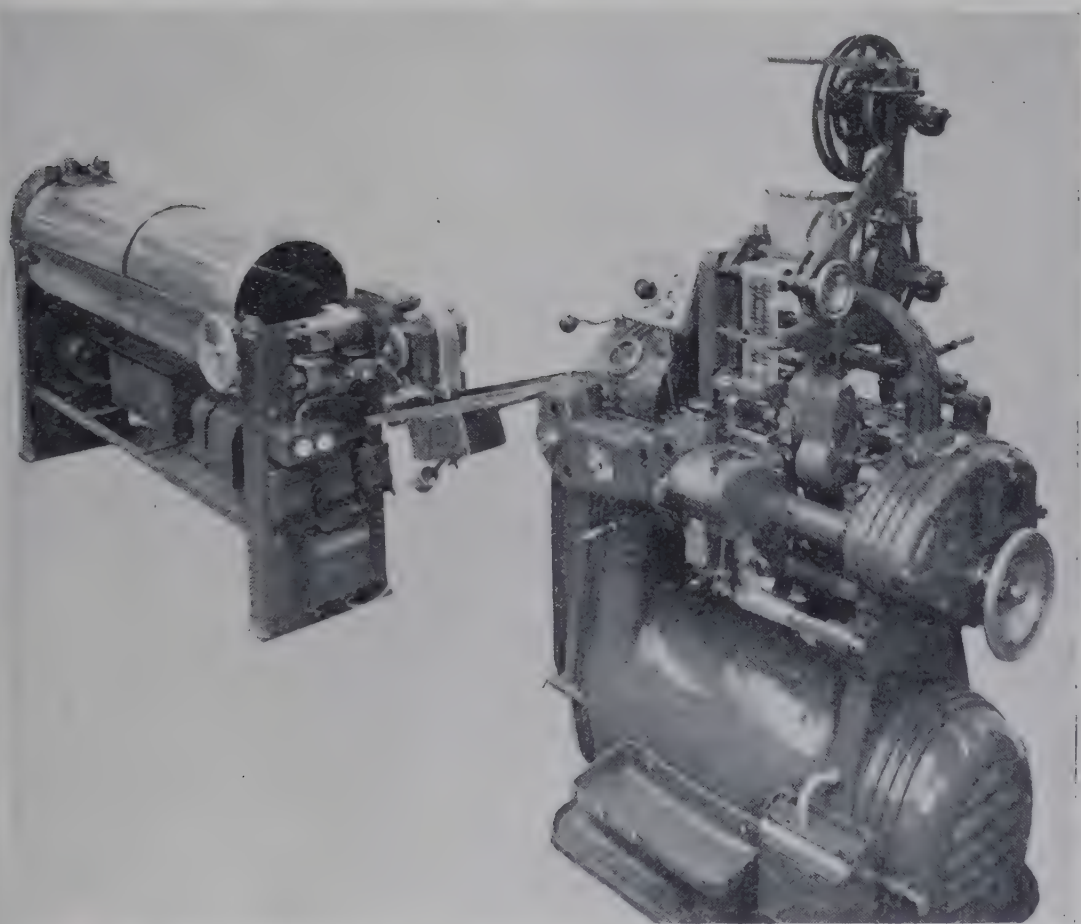

FIG. 6.—HIGH-SPEED F.W.T. PLASTIC TOFFEE CUTTING AND WRAPPING
MACHINE WITH AUTOMATIC BATCH ROLLER.
Output 650 wrapped pieces per minute.
(Rose Bros. (Gainsborough), Ltd.)

and wrap" work as the rougher handling these batches get
tends to induce grain.

C. FAT.—This must have a melting point below body tem-
perature in order that no unpleasant "tallowy" taste is to
be found. Commercial lecithin is often used to disperse
the fat properly but with a reasonable amount of milk
present this is unnecessary as the milk acts as an emulsifier.

D. MILK.—If dried milk is used it must be reconstituted and
passed through an emulsifier to give a smooth product.
The most usual form is condensed milk, either full-cream
or skimmed. It is the caramelisation of the milk solids
which gives the characteristic flavour. However, this can
easily go too far, especially with high boiled toffees. High
steam pressure and pans with contra-rotating stirrers should
be used.

Fudge

Fudge is nothing more than caramel in which graining has been
deliberately induced. The usual practice is to boil a batch as for
caramel and then induce crystallization by adding fondant. It is
best to cool the batch to 180° F. to 200° F. before adding the
fondant otherwise the crystallization may be very coarse and
appear as white blotches on the surface. Frappé is sometimes
added to lighten the texture.

NOUGAT AND MARSHMALLOW

Both these confections rely on aeration for their texture. The
aerating agents used are gelatin and egg albumen. In producing
nougat from gelatin, sugar and starch syrup are boiled to
a high temperature (around 270° F.) and poured into a beater,
soaked gelatin is added and the mass beaten till light and stiff.
Flavour and sometimes colour, nuts and fruit, are added. The
mass is levelled on a slab and cut when cool. For hard, chewy
nougats the proportion of starch syrup is high—usually 50 per
cent or more. For short nougats the porportion is reduced and
sometimes icing sugar is added after beating.

A similar procedure is followed with albumen except for one
major modification. Because albumen will coagulate at the
temperature of the syrup, the albumen is first whisked with water

until stiff; the boiled syrup is then carefully added, at slow speed, and stirring is stopped as soon as it is mixed in. A little melted fat (1 to 2 per cent) may be added right at the end in order to aid cutting. Many modifications are made—such ingredients as honey, invert sugar, milk powder, malt powder, cocoa powder, sometimes being added.

The manufacture of marshmallows follows the basic nougat method, except that the products contain less glucose and more moisture.

LOZENGES

These are made from icing sugar and gum arabic and/or gum tragacanth mucilage, which are mixed to a smooth paste in a dough mixer, with a small proportion of soaked gelatin. In the cheaper grades wheat flour or farina is used partly to replace the sugar. Flavour is added and the batch turned out into a lozenge cutting machine. The water content of the paste is about 8 per cent; care should be taken that a standard figure is obtained, otherwise variations in cutting performance will result. The cutting machine extrudes the paste in sheet form and then stamps out the individual lozenges.

These are then placed in a warm room to dry out to less than 2 per cent moisture. The stove temperature must not exceed 100° F. otherwise volatile flavours, especially peppermint, will be lost. The relative humidity of the air is not so important since lozenges will lose water in air with relative humidities of up to about 85 per cent. The main consideration is that the air be kept moving. After drying the lozenges are brushed and packed.

D. W. J.

CHAPTER 3

CHOCOLATE MANUFACTURE

In a single chapter it is possible to deal only with the basic principles involved in the manufacture of chocolate and methods of process control.

The consumer is familiar with chocolate as a solid; but in the factory it is fluid for the greater part of the manufacturing process, its temperature being maintained above the melting point. As a fluid, it consists of a continuous fat phase in which are dispersed finely ground sugar, cocoa solids and in the case of milk chocolate, milk solids. Small amounts of flavour and other materials to impart particular properties may also be included.

RAW MATERIALS

Cocoa

The cocoa bean, the seed of Theobroma Cacao, is a tropical crop. Botanically there are two main groups, the purple seeded Forastero which supplies most of the world's cocoa, and the white seeded Criollo, a less abundant type which produces a mild flavoured high quality cocoa. A number of Forastero varieties are recognised and in some producing areas there is much hybridisation among them and with the Criollo group. The flavour of a cocoa depends on the kinds of tree and the treatment of the harvested crop, and is therefore characteristic of the producing area from which it takes its name.

Table I shows the world production of raw beans for the crop year 1952–53.

Commercially, it is usual to distinguish between "fine" or flavour cocoas and bulk grades. Trinidad and Arriba are examples of the flavour type while W. African is an example of bulk cocoa. Best flavours are obtained by blending "fine" cocoas with each other or with bulk grades.

The fruit of the cocoa tree is borne on the trunk and main branches. It consists of a pod containing pulp in which the

TABLE I

PRODUCTION OF RAW COCOA BY COUNTRIES, 1952–53, IN THOUSAND LONG TONS[1]

Gold Coast . . .	247	Venezuela . . .	17
Nigeria	107	Fernando Po . . .	17
Brazil	95	San Thome . . .	10
Ivory Coast . . .	58	Colombia . . .	14
French Cameroons . .	52	British West Indies . .	14
Dominican Republic .	28	Mexico, Costa Rica	
Ecuador . . .	28	etc. . . .	43

seeds are embedded. The ripe pods are selected from the tree and the seeds or beans are scooped out, piled in heaps which are covered with leaves or in perforated boxes and fermented. In this way the flavour substances are modified in such a manner that the desirable chocolate aroma is produced on subsequent roasting. The collection and preparation of raw cocoa is a craft perhaps best exemplified in the plantations of Trinidad. Today there is a danger that the art will be lost. Under-fermented cocoa gives rise to a harsh astringent flavour while over-fermented cocoa gives a chocolate lacking in flavour.

Bulk cocoa of the Forastero type is graded by means of a "Cut Test" which shows the adequacy of fermentation and the quality of the beans. The test is used in commercial grading and as a factory control test. The beans, suitably sampled, are cut longitudinally across the cotyledons which are inspected for colour, texture and freedom from mould or insect attack. Unfermented dried beans are slatey with a dense cheesy texture, fully fermented beans are brown and friable with spaces between the cotyledons and the under-fermented beans are purple in colour and vary between these extremes. The quality of fine cocoas, on the other hand, is best judged by actual tasting.

The natural variations between different lots of the same kind of beans are balanced out by blending so that a uniform flavour and colour is obtained. Only the cotyledon is used for chocolate manufacture where it is usual to roast the beans. After separation, the shell-free cotyledon is known as "nib." The approximate composition of blended, roasted nib is given in Table II.

c

TABLE II

APPROXIMATE COMPOSITION OF ROASTED NIBS

	per cent
Fat	54
Water . . .	2
Protein . . .	11
Alkaloids . . .	1·5
Carbohydrate . .	9
Fibre	3
N free extract . .	8
Tannins . . .	6
Ash	3
Organic Acid . .	2·5

Sugar

The sugar which may be either cane or beet should be of high purity.

Cocoa Butter

Cocoa butter, the fat of the cocoa bean, has two properties which make it outstanding; its particular melting range and, compared with other fats, its exceptional stability.

Strongly flavoured chocolates can be made by the simple mixing of nib and sugar but modern recipes require a smaller proportion of nib with the addition of cocoa butter. High quality fat with a good cocoa flavour is obtained from the associated cocoa powder industry in which a portion of the fat is expressed from ground nib.

Adulteration of cocoa butter with substitute fats can be detected by determining the chemical and physical constants of the sample: but in small proportions, and especially in the case of Illipe type butters, it cannot readily be detected.

Milk

Milk may be obtained in a variety of forms varying from fresh milk at about 12 per cent solids to milk powder at 97 per cent total solids. Intermediate in this range are liquid condensed milk, to which sugar has been added, at about 74 per cent total solids and "block" condensed milk at 90 per cent total solids.

In all these forms the composition of the milk solids

FERMENTED
DRIED BEANS

|

CLEAN

|

ROAST

BREAK AND WINNOW

NIB

GRIND

SUGAR
FLAVOUR
LECITHIN
COCOA BUTTER
MILK

COCOA
MASS

MELANGEUR

|

REFINE

COCOA BUTTER
LECITHIN

CONCHE

CHOCOLATE

TEMPER

MOULD   ENROBE

FIG. 1.—(*right*)
PROCESSING OF CHOCOLATE.

should be that derived from fresh full cream milk. Milk fat readily develops off-flavours on storage and that in milk powder is particularly susceptible. The stability of milk fat is improved after blending it with cocoa butter.

Flavours

A wide variety of flavours is used for chocolate including spices, honey, coffee and vanilla. The last of these is the most common, either as an extract of vanilla pods or as synthetic vanillin.

Commercial Lecithin

Lecithin is a naturally occurring surface active agent which is produced chiefly from soya beans or groundnuts and is used in chocolate to save fat. Commercial lecithin is purchased principally on the basis of its acetone insoluble matter (usually 60 to 65 per cent) and freedom from unpleasant taste and smell.

The relative amounts of these ingredients are shown in Table III.

TABLE III

APPROXIMATE COMPOSITION OF PLAIN AND MILK CHOCOLATE

Ingredient	Plain	Milk
Blended Nibs (Shell-free beans) .	30–50%	10–15%
Sugar	40–50%	35–45%
Cocoa Butter . .	10–20%	15–25%
Milk Solids . . .	—	15–25%
Flavour . . .	Trace	Trace
Lecithin (Commercial) .	0–0·3%	0–0·3%
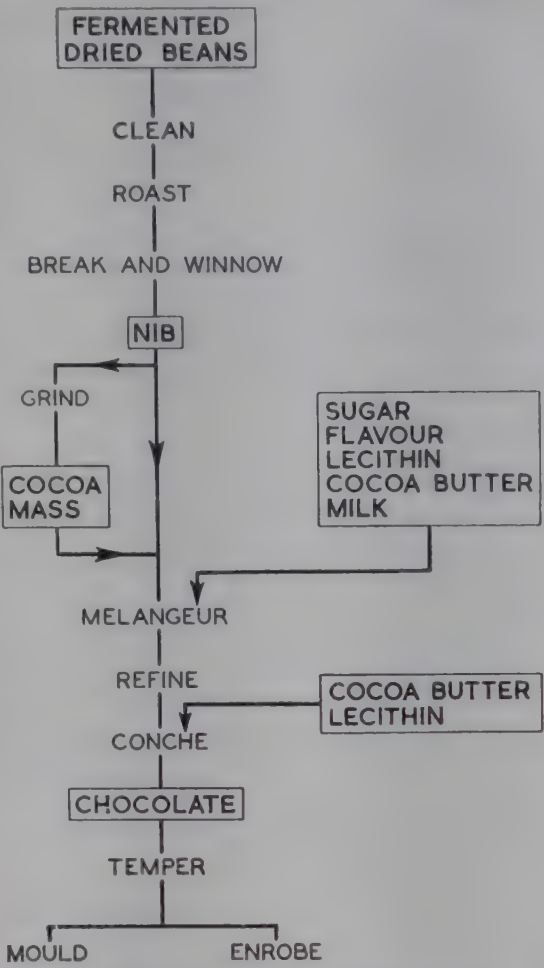
PROCESSING

An outline of chocolate processing is given in Fig. 1. The various stages will now be briefly described.

Roasting

Cocoa is roasted in order to develop the characteristic aroma and flavour of chocolate and to drive off unpleasant volatile substances. At the same time, the nib is dried and the shell loosened.

Roasting may be carried out either as a continuous or as a batch process. The advantage of batch roasting is that the variation in individual lots of beans can be catered for. Batch roasting is carried out in a rotating drum, which is directly heated or by passing a blast of hot air through the beans. Under these conditions the operation is controlled by a craftsman who samples the beans during the process and judges the correct point at which to stop. The beans are then discharged on to a sieve and cooled by blowing air through the perforations.

Continuous roasters are of the drum type, in which the beans travel slowly along a horizontal drum through which hot air is passed, or of the tower type in which the beans fall down a baffled tower up which hot air passes. Steam roasting and vacuum roasting are also practised although the latter method is alleged to give a less intense flavour development.

Breaking and Winnowing

Shell is removed as thoroughly as possible because it impairs the flavour and is difficult to grind.

The brittle, roasted beans are cracked between toothed rollers which can be adjusted to the size of the beans and the fragments are passed through a series of rotating cylindrical sieves. Each sieve fraction meets a counter current of air, the velocity of which is adjusted to effect the separation of shell and nib in that fraction. By operating on a small sieve fraction containing nib and shell fragments of comparable size, it is possible to obtain an almost complete separation. The shell and nib appear as size fractions on either side of the machine. Efficient winnowing can yield nibs containing about 1 per cent of shell. The separation of the smaller fractions of nib and shell is less complete.

The recovery of usable nib is of the order of 80 to 83 per cent of the raw cocoa. Cocoa and chocolate residues are used as a source of theobromine. The use of residues as a cattle feed supplement has fallen out of favour.

The amount of shell in nib is most easily and certainly estimated by physical separation. A sample of the nib is hand-picked and the percentage of shell determined by weighing. Methods which are suitable for chocolate are via the estimation of fibre[2] and the determination of pectic acid.[3]

Grinding

Cocoa nibs and crystal sugar may be mixed in the melangeur pan, but an alternative procedure involves grinding the sugar in an impact mill and grinding the nibs in a chocolate liquor mill. The latter process is claimed to give a more thorough mixing of the blend of bulk and flavour cocoas.

Grinding may take place between horizontally turning stones which are arranged in series so as to give two or three consecutive grinds.

More modern methods use a series of vertical discs, a series of rollers, or a combination of the two. During the process the fat cells are ruptured and the heat of friction liquefies the mass. The resulting liquor or "cocoa mass" may be used directly in the melangeur pan, "blocked-off" for storage or supplied to a hydraulic press for cocoa powder manufacture.

Melangeuring

The aim of melangeuring is to mix the ingredients and to produce a homogeneous paste suitable for feeding to the refiners.

The melangeur pan consists of a pair of granite rollers suspended in an over-carriage and riding on a horizontal, circular, rotating bed of steel or granite which may be heated. Diametrically opposed ploughs continually return the paste to the rollers. The appropriate amount of cocoa is charged into the pan either as nib or cocoa mass together with sugar and sufficient cocoa butter to give a suitable working consistency. Flavour and some lecithin may also be added. If milk chocolate is being manufactured the milk is introduced at this stage. Milk powder or block condensed milk can be added directly, but liquid

condensed or fresh milk requires preliminary processing in order to eliminate most of the water.

Refining

The mixture from the melangeur pan may contain quite coarse particles, and these are reduced in size by passing the paste through refining rolls where it is ground to give a smooth texture. The paste may be given a preliminary treatment on a three-roll refiner, but five-roll machines are widely used. The five horizontal rollers which are made of chilled cast iron are highly polished and water is passed through the hollow core to prevent over-heating and expansion. Successive rolls rotate in opposite directions and there is a progressive increase in speed and a reduction in the clearance between the rolls. The paste is fed to the nip of the first pair of rollers where the oversize particles are crushed, transferred to the second roller, passed through the second nip and so on, to receive four separate grindings.

Before refining the material is fairly fluid but it is scraped off the top roller as a dryish flake. The change in consistency is mainly caused by the increase in surface area of the pulverised solid particles which require more fat to coat them.

The smoothness of chocolate is most directly assessed by tasting but the limitations of such a subjective test are obvious and it is usual to use a particle size method to assess the "fineness" of chocolate. According to Jensen,[4] it is doubtful whether the palate can detect sugar particles less than 50μ in diameter but a small proportion of particles exceeding 100μ in diameter may give an impression of roughness. Modern refiners can yield high quality chocolates in which 90 per cent of the particles are less than 20μ.

Sieving tests have been applied to dispersions of chocolate in petrol but they have not been developed to give information about the distribution of particle sizes below the diameter of the coarsest particles. The size distribution can be obtained from microscopic examination but the procedure is tedious.

Mason has applied a lycopodium count method to obtain a *micro-value* for chocolate which is a single numerical expression for the fineness of chocolate.[5] For control purposes the most suitable microscopic method is to compare a film of the test chocolate with a standard film by projecting on a screen.

The objection to all microscopic methods is that the sample is small and possibly not representative of the bulk. Recently progress has been made in the measurement of size distribution in fine powders[6] and the application of the Andreasen sedimentation method[7] to chocolate has been described.[2]

Conching

Chocolate from the refiners is usually conched before being used. The original conche, so-called because of its shell-like shape, consisted of a tank with a bed of granite on which two heavy rollers moved backwards and forwards. The traditional long conche is a simple development of this.

Conching consists of subjecting the refined chocolate mass to the to-and-fro motion of the rollers for varying lengths of time. Plain chocolate may be conched for as long as 7 days, but this is impracticable for bulk production and 2 to 3 days is more usual. Milk chocolate requires a shorter time. Very wide temperature ranges are used, 120° F. to 195° F. for plain and 110° F. to 145° F. for milk. During conching, cocoa butter is added to bring the chocolate to a suitable working consistency and additions of volatile flavouring and of lecithin may be made. Subsequent to conching further cocoa butter is added according to the purpose for which the chocolate is required.

The chemical and physical effects of conching are the subject of much speculation. Among the claims which have been made are the following:

Separation of the agglomerates into very fine particles.

Emulsification of the particles in the fat.

Removal of air, moisture and acid materials.

Development of flavour by a wide variety of reactions.

Increased fluidity.

Rounding off the corners of the larger sugar crystals to give greater smoothness.

Oxidation of some cocoa butter to give enhanced resistance to bloom.

It would be difficult to substantiate some of these claims, but there is no doubt that conching produces a marked improvement in chocolate. Many alternative machines have been designed to modify and shorten the process.

CHOCOLATE FLUIDITY

For maximum production efficiency, and uniformity of quality, it is essential to have chocolates with uniform fluidities. Although an experienced craftsman may be able to judge fluidity subjectively, it is necessary to use a more objective method for the control of bulk production.

Methods of Control

Many instruments have been used to measure the fluidity of chocolate. Among them may be mentioned the use of sinkers, capillary instruments, Redwood types of viscometer employing the measurement of flow through a wide orifice,[8] and various types of rotation viscometer. Of the last type, the MacMichael viscometer has been widely used and recommended as a standard instrument in America.[9] It is generally accepted that chocolate is not a simple fluid, but is of the nature of a "Bingham" plastic material[10] and possesses a small yield value. It is necessary therefore, either to specify the flow properties completely, or to ensure that the fluidity test bears some relationship to the behaviour of the chocolate as it is used. The usual method of control is to manufacture chocolate with a slightly low fluidity, and then to make a final adjustment by the addition of cocoa butter.

Factors Affecting Fluidity

During processing, a number of factors affect fluidity, and hence the fat content of the finished product. Small quantities of lecithin produce an increase in fluidity, so that lecithin may be used to a limited extent to replace cocoa butter.[11] The reduction in particle size on refining will cause a decrease in fluidity, and a proper balance must be maintained between the requirements of smoothness on one hand and fluidity on the other. The addition of small amounts of water to chocolate causes a noticeable decrease in fluidity, and it is probable that inadequate moisture elimination, especially in the case of milk chocolate, will have an adverse effect. In general, the more agitation that a chocolate receives without a reduction in particle size, the more fluid the product, although the effect of agitation is less marked in the case of chocolates which contain added lecithin.

MOULDING AND COVERING

The processes used for producing finished chocolate goods may be classified as moulding and covering. Moulding is carried out on many different machines, but all have the common feature of the deposition of a measured quantity of chocolate into a mould, whether the product is a solid tablet, a centred tablet or a hollow object; in the latter case the mould is placed in a spinning device. Covering may be carried out by hand or more usually by passing the centres through a curtain of liquid chocolate on an "enrobing" machine.

Whatever the process, chocolate must be cooled from the temperature at which it is liquid until it solidifies and finally reaches the normal atmospheric temperature. This cooling process must be controlled, otherwise the resulting solid product will be likely to have a grey, unattractive appearance devoid of gloss, a coarse granular texture with a weak fracture and a poor breakdown in the mouth. The first stage of controlled cooling brings the chocolate into a suitable condition for moulding or covering and is known as "tempering." The second stage involves passing the moulded chocolate or covered centres through cooling tunnels in which the temperature conditions are carefully controlled.

Tempering

In this process, a small fraction of the fat is induced to crystallise and the grains of solid fat are distributed evenly through the bulk. The "grain," as it is known, acts as seed to promote the proper crystallization of the remainder of the fat. The art of tempering lies in the production of a grain which will result in the desired properties in the finished product. The temperature of tempered chocolate will generally be in the range 86° F. to 92° F.

The principle of tempering is exemplified by the hand method in which chocolate, at about 95° F. drawn from a vessel, is cooled by spreading it on a cold surface. The solidified chocolate is then scraped off and mixed with the overlying liquid chocolate. The operator judges the temperature and consistency of the mass and endeavours to maintain it at a constant temperature. Any tendency for the grain to disappear is counteracted by

further cooling while a tendency for the grain to coarsen is checked by adding warm untempered chocolate.

In bulk, chocolate is tempered in batches in a water jacketed kettle equipped with scrapers and stirrers, or by continuous tempering machines which pass the chocolate through the appropriate temperature cycle over water-cooled rollers or down a spiral ribbon-shaped space in a water-cooled cylinder. Once the conditions for tempering a given chocolate with a particular piece of equipment have been determined, they can be specified for repeated use. In order to avoid supercooling and to check the tendency of the grain to coarsen, it is usual to cool the chocolate below the desired temperature and then to bring it back by cautious reheating. There is a decrease of fluidity on tempering, and a well-tempered chocolate should have the maximum fluidity compatible with the other requirements.

Radically different methods of tempering which have been suggested in the literature are by the cautious re-melting of blocked-off chocolate[12]; the addition of solid chocolate flakes to a cooled but untempered bulk; the addition of untempered chocolate to a grained mass. An application of the last method, particularly suitable for enrobers is "drip-feed."[13] The temper of the circulating chocolate in the enrober is held constant by the addition of untempered[14] or under-tempered chocolate[15] at the same rate as it is being used.

The state of temper can be assessed by the use of cooling curves. A temperature-time graph is obtained when cooling a small sample of the chocolate, and with a little experience, it is fairly easy to relate the shape of the curve to the behaviour of the tempered chocolate. Conditions ranging from cooling with dry ice and acetone[16] to cooling in the factory atmosphere[17] have been proposed. A "Bob Test"[18] has also been described for chocolate of standardised viscosity. The weight of tempered chocolate left on a metal bob is determined after dipping and draining and compared with the weight of untempered chocolate which remains under similar conditions.

Cooling Tunnels

After depositing or covering with tempered chocolate, the goods pass through a cooling tunnel which is so arranged that the minimum temperature is near the middle. Intense cooling

must be avoided, and the time in the tunnel will depend on the dimensions of the individual pieces. The final temperature on emerging from the tunnel should be above the dew point of the atmosphere.

STORAGE PROPERTIES

Chocolate is a remarkably stable material, which under normal conditions will keep indefinitely and this in conjunction with a high nutritive value makes it valuable not only as a confection but also for emergency rations.

High relative humidities and temperatures should be avoided. Above about 80 per cent R.H. sugar may dissolve on the surface and when the humidity falls again the syrup dries with the hard rough surface characteristic of "sugar bloom." The same defect will arise if chocolate is cooled below the dew point so that water can condense it. Exposure to high temperatures may cause the chocolate to melt, and on re-solidifying leave a light coloured surface with no gloss and a rough texture.

"Fat bloom" may appear as a soft greyish blemish on the surface of chocolate after several weeks storage. It is particularly liable to occur on the covering of oily or nutty centres, the oil from which diffuses outwards, dissolving cocoa butter and depositing it on the surface.[2] A more general cause however may be the existence of polymorphism in cocoa butter, and it is found that fat bloom can be partially inhibited by careful tempering and cooling. Numerous bloom inhibitors have been proposed and include fatty acid esters of higher alcohols[19] and small amounts of hardened palm-nut or hardened arachis oil.[20] Attempts have also been made to modify the cocoa butter itself.[21]

Saponification of fat is liable to occur in chocolate in the presence of moisture and has been observed when coconut and palm kernel fats were used as anti-bloom agents.[22]

Cocoa butter does not readily develop oxidative rancidity. One reason is that cocoa contains natural antioxidants and the effect is so marked that chocolate liquor will protect other fats from this sort of deterioration.

Insect Infestation

Wherever foodstuffs are stored and handled there is a great danger of infestation by insects originally brought in with the

raw materials. Cocoa products are liable to damage caused by the larvae of the cocoa moth. A continuous effort is necessary on the part of all concerned in the handling of cocoa and chocolate to keep this pest under control. Suitable control measures involve the education of operatives, coupled with frequent inspection, absolute cleanliness, spraying and fumigation.

<div align="right">A. N.</div>

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CHAPTER 4

JAM MANUFACTURE

THE factory production of jam has developed from an originally domestic method of preserving fruit for use in the winter months and for preparing an attractive confection. The application of scientific principles has enabled jam to conform to certain standards, to be a uniform product with a reliable keeping quality, withstanding the shocks of transport and distribution.

SCIENTIFIC PRINCIPLES

Jam is essentially a gel containing fruit either in a whole or pulped condition. The gel is formed by pectin in the presence of sugar and acid and the pectin is derived from the fruit. Most plant tissues contain pectinous substances or hemicelluloses. These act as the cement which binds the cells of the tissues together and they have been given the name of proto-pectin since from them is obtained the substance, pectin, which is soluble in water and is the setting agent in jam. Proto-pectin reaches its maximum quantity in fruits just before picking time. During ripening it is changed by enzymes into pectin and pectinic acid and finally into pectic acid. It can be readily understood how the fruits become softer and more juicy as the adhesive between the cells breaks down. The only substance which enters into jelly formation is pectin and it is therefore of paramount importance to obtain and preserve as much of this from the fruit as possible. It is obviously essential that the fruits should, in the first instance, be in sound condition and not over ripe so that when the fruit is cooked in the factory as much pectin as possible is obtained. As the amount and quality of the pectin obtained is extremely variable it is common practice, in order to produce a uniform product, to add during manufacture, pectin extracts of standardised strength, obtained from apples or citrus fruits.

Pectin, as it is formed from protopectin, dissolves in water and becomes negatively charged due to the presence of the fruit

cids. The addition of a large quantity of sugar upsets the pectin-in-water equilibrium and the pectin conglomerates and forms a network of fibres enclosing the liquid, converting it into what is known as a jelly. This gel formation only occurs within a narrow range of hydrogen ion concentration from 2·5 to 3·45, and is best at pH3. The sugar concentration also has an effect on the rigidity of the gel and the optimum is about 67·5 per cent although with increased pectin and acid, jellies can be formed with 60 per cent sugar. Normally, 0·5 per cent to 1·0 per cent pectin is used in jam manufacture.

In the right proportion pectin, sugar and acid will form a gel in the cold. This is not possible with cooked pulp without driving off a considerable amount of the water present. This is done after the addition of sugar, by boiling in steam jacketed pans. Conditions are arranged to make the period of boiling as short as possible so that the minimum amount of pectin is destroyed by heat and, with adjustments made to give the correct pH value, the end of the boiling is determined by the rise of the soluble solids present in the jam to 65 to 70 per cent. After partial cooling, this product can be poured into jars and allowed to cool to room temperature by which time it will be set into the familiar, more or less translucent jelly.

FRUIT AND ITS PREPARATION

From the underlying principles of jam making it will be seen that the type or variety of fruit used and its condition are important considerations in jam making. Other factors are suitability for transport and in preparation and also pectin content. It is wrong to suppose that any variety is good enough for jam. Colour, size and flavour all have their effect on the finished jam. The worldwide reputation enjoyed by jam made in the United Kingdom, owes something to the use of choice fruits which are not available anywhere else in the world. The excellence of British strawberries, raspberries, blackcurrants and gooseberries is apparent in the high quality preserve prepared from them and judicious use of varieties can play an important part in producing the best.

Jam fruits can be divided into two groups. The first are the soft fruits of delicate flavour containing little pectin. The

others are mainly hard stoned fruits which are poorer in flavour but rich in pectin. Strawberries, raspberries, cherries and blackcurrants are in the first group while plums, apples, bitter oranges and gooseberries comprise the second. The average composition of a number of fruits is given in Table I.

TABLE I

Variety	Insoluble Solids	Soluble Solids	Total Solids	Total Sugars	Acid as Citric	Pectin as Crude Calcium Pectate
Gooseberries –	2·61	8·45	11·06	3·51	2·22	0·81
Strawberries –	2·14	8·98	11·12	5·48	0·93	0·53
Raspberries –	6·17	7·98	14·15	3·58	1·73	0·53
Blackcurrants –	5·69	14·25	19·94	6·44	3·48	1·08
Victoria Plums –	1·13	12·63	13·76	7·43	1·64	0·81
Yellow Plums –	1·03	10·80	11·83	5·69	1·47	0·80

Strawberries

Strawberries are of better quality in England than in any other country in the world and are in great demand for jam making. Many problems, however, are encountered in converting them to jam. The varieties mostly grown are the early cropping Royal Sovereign and the late Huxley or Brenda Gautry. Both varieties are sensitive to climatic conditions and prone to virus disease although resistant varieties have been developed. Both varieties produce firm well coloured fruits which travel well. This latter is an important consideration when fruit may have to be transported long distances from the field to the factory. Strawberries are easily bruised and then the juice rapidly drains away, involving heavy losses in weight and flavour. For this reason they should be picked just before they are fully ripe.

One of the major problems in handling strawberries is the removal of the stalk and plug. This is an operation which can only be done by hand and is usually performed by women, sometimes on a site near the field but usually in the jam factory. Opportunity is usually taken at the time of plugging to remove all unsound fruit. The removal of the stems and plugs of Royal Sovereign strawberries is a comparatively clean and easy operation but with the Huxley variety it is difficult and often impossible to remove the plug. Plugged strawberries should be processed immediately as they quickly deteriorate. It is, however, a

great mistake to pulp or cook strawberries on stalk. This practice will always adversely effect the flavour and colour of the final jam. Any dirt or grit should also be removed and this can be done by spraying with water or if the fruit is in baskets, by swirling in a tank of clean water.

Where it is found necessary to transport strawberries over long distances from the large growing areas to the big towns, it is an advantage to keep the fruit as cool as possible. If packed in totally enclosed trucks or wagons, blocks of solidified carbon dioxide can be used to reduce and maintain the temperature at about 40° F. for the period of the journey.

Raspberries

Raspberries are almost as popular as strawberries because of their attractive flavour. Lloyd George and Norfolk Giant are the two most important varieties. They should be picked carefully without plugs when they are fully ripe and converted to jam as quickly as possible. Sometimes raspberries will develop mould growth within a few hours if they are moist and the weather is warm. Solid carbon dioxide can again be used if fruits are in good condition and it is necessary to store or transport overnight. For transport purposes casks are preferable to baskets as they prevent wastage of juice. Under all circumstances they should be handled as little as possible.

Blackcurrants

Blackcurrants are appreciated for their fine flavour and high nutritional value. Careful treatment will preserve the distinctive flavour but if it is required to retain the Vitamin C in the finished jam it is essential to avoid contact with all other metals except stainless steel. Copper which is the commonest metal for the fabrication of jam making plant is very active as a catalyst in the oxidation of Vitamin C.

Most varieties of blackcurrants are suitable for jam-making but the best is probably "Baldwin." The fruit usually arrives at the factory on stalks which can either be removed by hand, using a table fork to free the berries or by a strigging machine. This machine is simply a vibrating frame set at a shallow angle over which is stretched longitudinally a set of wires which are

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spaced close enough to prevent the fruit passing through but not the stalks.   Strigging is more efficient if the blackcurrants are previously chilled so that they are hard.

Gooseberries

Gooseberries can produce a very excellent jam but there is only a small demand for this preserve.   The most important consideration for jam-making is the stage of ripeness and they should always be picked when pale green.   Ripe gooseberries lack the delicate flavour of the greener fruit.   They produce a muddy coloured jam and the skins are tough.   Before cooking, the tops and tails must be removed.   This is carried out in a snibbing machine in which the gooseberries are thrown against the emery-lined internal walls of a cylinder.   Water sprays help to remove the waste material and also to wash the fruit.

Plums

Plums are very suitable for jam making as they contain both pectin and acid in plenty.   The jam has an attractive sharp flavour which opposes the excessive sweetness.   In some years there is a glut of plums and they can usually be purchased cheaply. The most popular varieties are the Victoria and Pershore Egg plum with Monarch and Belle de Louvain slightly less so.   All these, except the Pershore Egg, are purple plums.   The Pershore Egg should be gathered when green and hard so as to obtain the maximum amount of pectin.   The size of plums plays an important part in their economic use since it is better to use a large plum with a small stone than a small plum with a large stone.   In this respect the Victoria is the best since it is the largest plum with only an average weight of stones.

Apples

Apples are never used alone in jam but they are very often a component of mixtures of fruit.   These mixed fruit jams are largely in demand by the bakery and catering trades.   Only the green and hard type of apples are suitable, not the soft kinds or those with pink flesh.   Because apples used for jam are usually culls unsuitable for sale in other ways, they require sorting and washing before use.

Citrus Fruits

Citrus fruits are imported into this country for the purpose of making marmalade. There are many different recipes and several types of this preserve so that it forms almost a separate subject. The majority of the oranges imported are the bitter Spanish type known as Seville, but some supplies come from South Africa and Palestine. Jamaica also sends orange pulp in barrels but it is usually paler in colour than pulp produced from Seville oranges. Because of the long sea journey that oranges have to undergo before reaching the factory, there is usually some loss from fruit being overripe and infected with mould particularly towards the end of a shipment. To minimise this loss, the cases of fruit should be stored in cool, well ventilated rooms. After sorting and washing, the fruit is quartered and the peel separated from the flesh or dummy by a specially designed machine. The peel is chipped or shredded before cooking in water, while the dummy is cooked by itself and sieved.

PRESERVATION OF FRUIT

Jam made from fresh fruit commands a higher price than that made from preserved pulp but very few factories are equipped to make all the fruit arriving during the very short harvesting season into finished jam. Even if it were possible, it is unlikely to be desirable to store such a large quantity of finished goods. Also by preserving the fruit, employment can be maintained throughout the year and in times of glut when fruit is cheap, enough pulp can be made to ensure against a bad harvest. The commonest method of preserving jam fruit in Britain is by the use of sulphurous acid, but freezing or canning is practised extensively in the United States. (Sulphurous acid H_2SO_3 is of course formed by dissolving sulphur dioxide gas (SO_2) in water. For simplicity in the text SO_2 is used instead of "sulphurous acid.") Citrus fruits, plums and blackcurrants are not seriously affected by storage in SO_2 but strawberries and raspberries lose their flavour and sometimes completely disintegrate.

Containers for Fruit Storage

Fruit can be preserved with SO_2 either in the raw or cooked

state. Strawberries, raspberries and blackberries are best treated without cooking, whereas those with skins such as plums, gooseberries, black and red currants, are usually cooked before preserving, as the storage in SO_2 tends to toughen the uncooked skins. The most convenient container for storage is the 40-gallon cask. Other methods such as glass containers or large concrete tanks are used, but none is so practical as a good oak cask. It is advisable not to economise on these containers by using soft wood barrels, as losses by leakage can be considerable. The SO_2 is used in the form of a 6 per cent solution in water which has a specific gravity of 1·030. The solution is added through the bunghole after the cask has been filled.

Sulphurous Acid as a Preservative

The main advantage of using sulphurous acid as a chemical preservative is that the bulk of it is driven off in the boiling process. Legal regulations require that there shall be not more than 40 p.p.m. of SO_2 in the finished jam. This is easily arranged if the preserved pulp contains from 1,000 to 1,500 p.p.m., except in the case of strawberries where the whole fruit tends to retain the preservative and the residual amount in the jam needs to be frequently checked. When pulp is purchased from a merchant it is to be expected that it will contain the amounts of water and SO_2 which were laid down in the war-time emergency regulations although the production of pulp is no longer controlled by Government Order.

In Great Britain, SO_2 is the only preservative used or permitted. It is almost always used in the form of a 6 per cent solution but occasionally Calcium Bisulphite is used in addition as it is believed to improve the firmness of whole strawberries. The regulations in South Africa and India concerning preservatives are the same as in Great Britain while Canada allows preservatives only in 2nd and 3rd grade jams if specified on the label. The United States permits the use of either SO_2 or sodium benzoate if declared. Australia, New Zealand and many continental countries prohibit the use of any preservative.

Casks for containing fruit pulp need to be thoroughly washed and then soaked for 24 hours in a 1 per cent solution of a caustic detergent. Finally they should be rinsed with clean water containing $\frac{1}{2}$ pint per gallon of 6 per cent SO_2 solution. If stored

for any length of time before use, about 5 gallons of this solution should be left in the cask. If the fruit is to be preserved in the uncooked state it should be carefully cleaned and prepared. For every hundredweight of strawberries to be placed in a cask, ½ gallon of fresh water is first put into the unheaded container. The fruit is then added and then a final ½ gallon of water per cwt. is poured in on top of the fruit. This procedure helps to mix the fruit and water as it is not possible to stir them up at all without breaking the berries. Raspberries and blackberries do not require any water. When the cask is full it can be headed up and placed on the roll. Three pints of 6 per cent SO_2 solution per 1 cwt. of fruit are then poured in through the bunghole and the cask rolled.

Preparation of Cooked Fruit Pulp

The preparation of pulp is a most important step in the process of jam manufacture and when it is necessary to cook the fruit for this purpose, many hundreds of pounds of jam may be spoilt by careless or faulty methods. The old-fashioned method was to use steaming barrels but these have been largely replaced by stainless steel pulping vats. These are large round vessels fitted with a lid. Near the bottom of the vat is fitted a single coil of steam pipe which is perforated every six inches to allow the steam to pass into the fruit. The outlet should be of the gate valve type that does not block easily. The cooking process, in which the fruit is steamed for 10 to 20 mins. should aim at bursting the skins but it should not be allowed to break up the fruit into a mash, destroying all the colour and flavour. The cooking process should also serve to preserve the pectin. The amount of fruit and water must be carefully controlled and also the weight of the finished batch so that the percentage of fruit in the finished pulp is known when it is used for jam making. The cooked pulp can be either put straight into casks, which should be done as hot as possible, or it can be passed through a sieving machine. This machine consists of an inclined cylindrical sieve inside which brushes revolve, pressing the pulp through the interstices of the sieve. The hot cooked pulp is poured in through a hopper and the sieved material collected in casks at the bottom of the lower end. Stones and debris are thrown out at the far end of the sieve. The casks of hot pulp are headed up
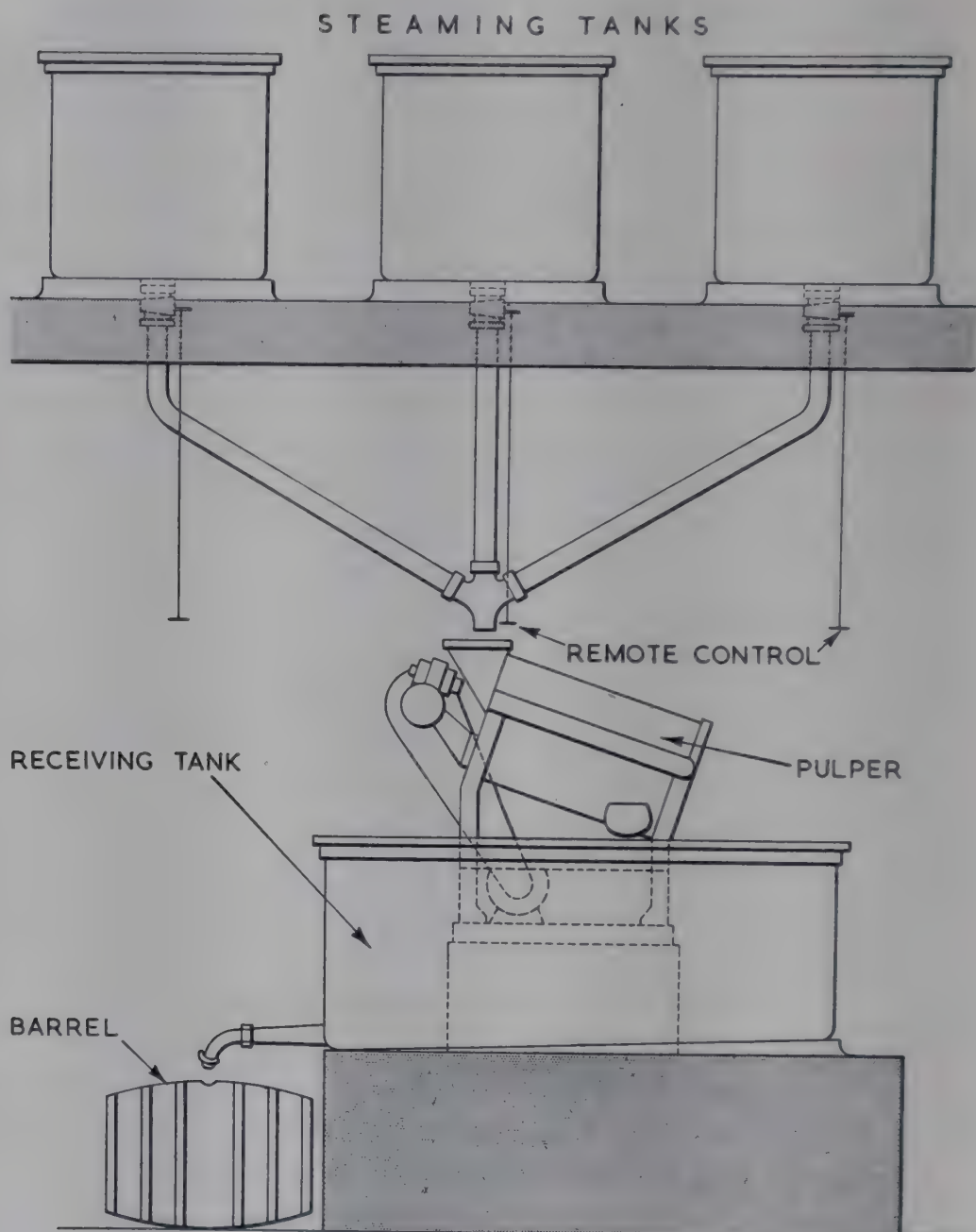
STEAMING TANKS

FIG. 1.—DIAGRAMATIC VIEW OF PULPING PLANT.

Fruit is cooked in the steaming tanks, passed through the sieving machine and collected in the Receiving Tank. Barrels can be filled from the Tank as required.

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and allowed to cool, lying on their sides for about 12 hours, when 3 pints of SO$_2$ solution are added for each 1 cwt. of pulp.

The storage of the casks of pulp needs careful attention. Standing in open yards is always bad, and cellars or well aired rooms, maintaining an even temperature are the best conditions. If that is not possible, simple sheds should be constructed.
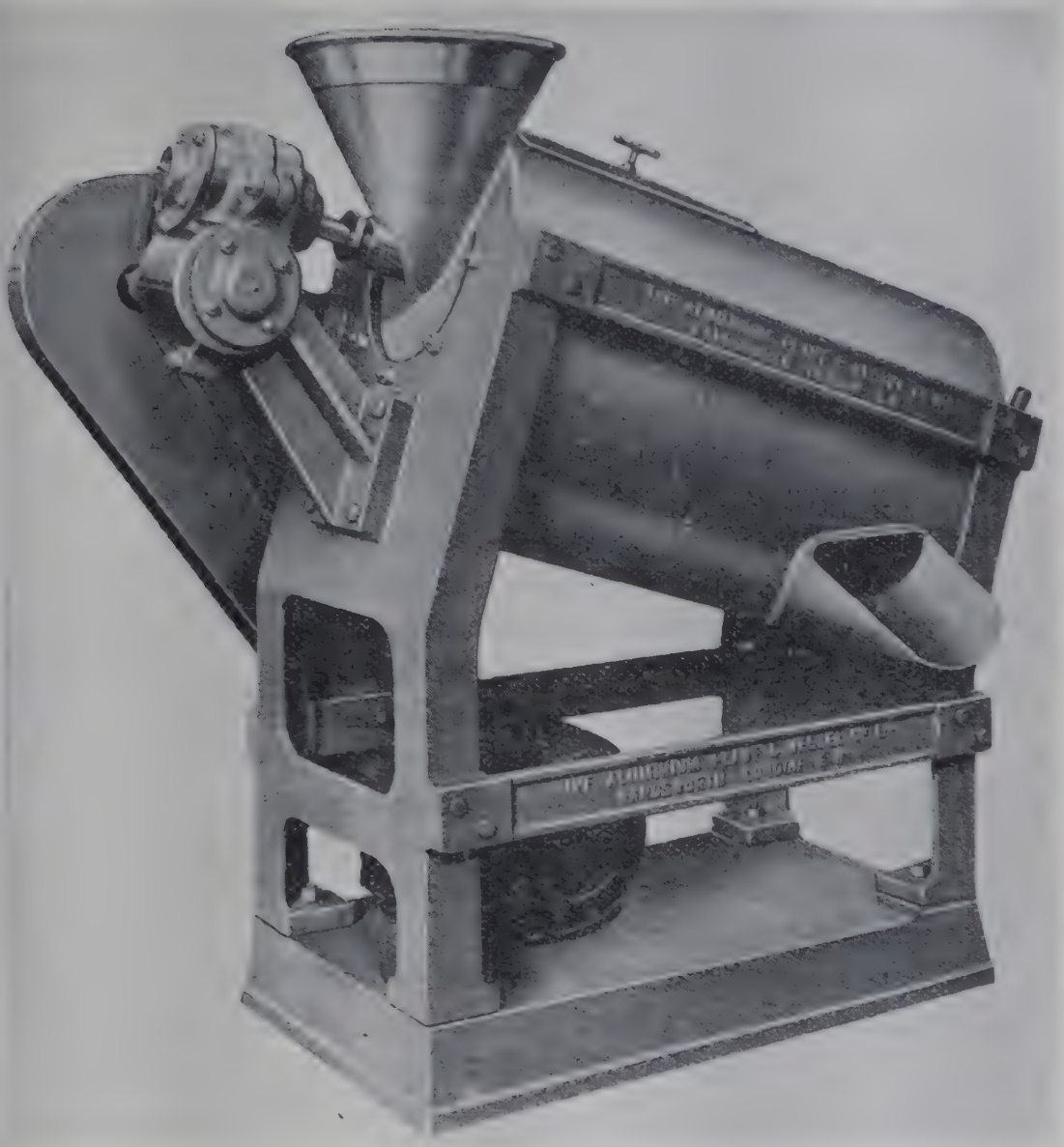
FIG. 2.—SIEVING MACHINE.

Fruit pulp is fed into the top hopper and the sieved material collected at the side outlet. All surfaces in contact with fruit are stainless steel and the brushes are easily changed.

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Regular, periodical, checks should be made on the SO_2 content and adjustments made if necessary. Each cask should be clearly identified and its history recorded in a card index, including the results of any inspection.

JAM BOILING

The actual boiling of jam can be carried out either in small open pans of about 40 gallon capacity or in large vacuum pans.

The former produces the higher quality jam and is common in Great Britain, while vacuum pans holding about a ton per charge are used in the United States where the emphasis is on a high rate of production.   Jam boiling in open pans requires considerable skill and experience and most factories have their own methods developed by their own skilled personnel.   There are, however, some basic rules which apply in all cases.

Basic Rules

1. THE BOILING TIME.—This should be as short as possible and should never exceed ten minutes.   Five to seven minutes is the optimum time.   Prolonged boiling reduces the setting power of the pectin and yields dull-looking, caramelised jam with poor flavour.   It also creates an excessive amount of invert sugar from which dextrose may crystallise out.

By carrying out the boiling process in small pans, the heating surface is large compared with the volume of the pan, and the steam bubbles formed on the metal surface rapidly rise to the surface and quickly evaporate.   Obviously the water which must be evaporated must be kept to as small an amount as possible.   The use of pectin extracts enables a set to be obtained without prolonged boiling.

2. CONTROL OF "SETTING" BY pH.—The "setting" of the jam is controlled by the pH of the boil.   All the ingredients, fruit pulp, sugar, pectin and water, affect the pH and further adjustments can be made by the addition of citric acid.   A good gel is only obtained when the pH lies between 3·0 and 3·2.

3. CONTROLLING THE DEGREE OF INVERSION.—The presence of acid, both in the fruit and added separately, causes inversion of the sucrose in which one molecule of sucrose is converted into one molecule of dextrose and one molecule of levulose which constitutes invert sugar.

$$C_{12}H_{22}O_{11} \; + \; H_2O \; = \; C_6H_{12}O_6 \; + \; C_6H_{12}O_6$$

$$\text{Sucrose} \qquad \text{Water} \qquad \text{Levulose} \qquad \text{Dextrose}$$
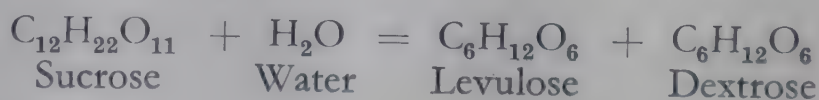
The degree of inversion must be controlled to give a final invert sugar content between 25 and 40 per cent.   If less than 25 per cent, cane sugar will crystallise.   If over 40 per cent, dextrose may crystallise or the "set" may be weak.

As already stated, the amount of invert sugar also depends on

the boiling time and can be controlled by the amount of water added to the boil.

4. SOLUBLE SOLIDS CONTENT.—It is essential in order to produce a jam of good quality and with satisfactory keeping properties, to boil to a soluble solids content of 65 to 70 per cent, depending on the method of finishing. War-time regulations stipulated a minimum of 68·5 per cent, and this still serves as a good end point for the jam. As the boil proceeds and water is evaporated, the temperature of boiling rises. This serves as a rough guide to the concentration of solids but it can only be approximate. Barometric pressure will alter the temperature and although correction can be made for this, the boil is not sufficiently homogenous for the thermometer reading to be an accurate indication of the total solids.

A more exact method is to use a refractometer. When the boiling temperature reaches 220° F., it indicates that the end point is approaching. From then on, tests can be made with the refractometer until 68·5 per cent solids is reached. The boil should then be rapidly turned out of the pan and quickly cooled to prevent any further increase in solids.

Variations between batches of fruit, together with the complicated interplay of these various factors, make it important to carry out trial boils daily or whenever a new batch of fruit is used. Adjustments can then be made in the recipe to give the required finished article.

Since it is required that the final jam shall contain 68·5 per cent soluble solids, it is possible, from a knowledge of the soluble solids in each ingredient, to calculate the theoretical out-turn. In the case of strawberry jam the following is an example.

Ingredient	Weight Used	Per Cent Soluble Solids	Weight of Solids
Sugar	65 lb.	100	65 lb.
Strawberry Pulp	34 lb.	9	3 lb.
Pectin	10 lb.	10	1 lb.
		Total	69 lb.

The 69 lb. of solids added to the pan will in the finished jam be 68·5 per cent of the whole. The out-turn will therefore be

100·75 lb. The final weight of each batch thus constitutes another check on the jam-making process. Each boil should be weighed and should not vary from the theoretical weight by more than 2 lb.

With these factors in mind a typical boiling procedure can be described as follows.

The Boiling Process

The sugar and water (about 1 gallon) are placed in the boiling pan and heated together to dissolve the sugar. In some plants sugar syrup is made in a separate plant, filtered, and pumped to
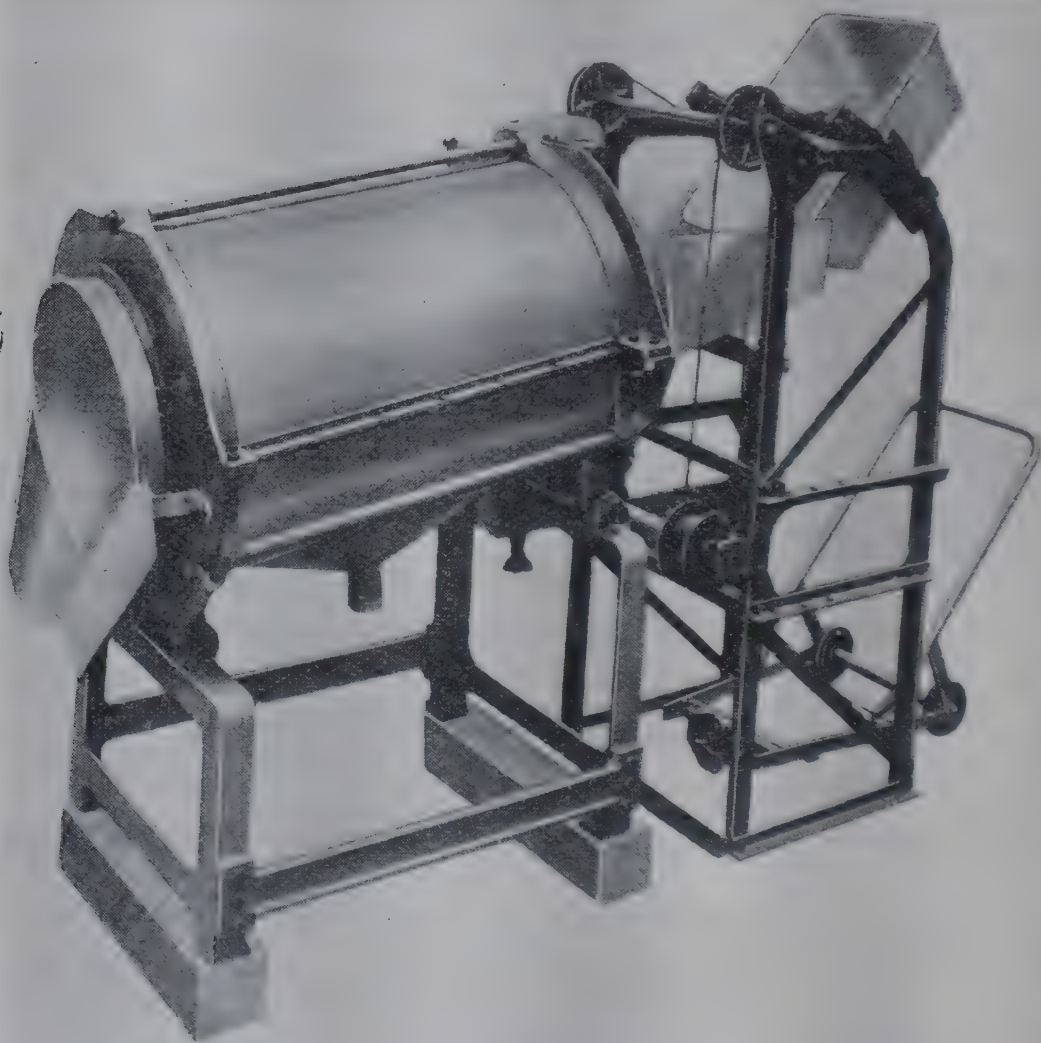

FIG. 3.—JAM COOLER.

The bogie-full of jam is lifted to the top of the elevator and automatically tipped down the central revolving cylinder which is watercooled on the outside.

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the boiling pans.   The fruit pulp and pectin are then added and the mixture vigorously boiled until the temperature, as recorded on a long-stemmed thermometer, reached 220° F.   The solids are then checked on the refractometer and as soon as they reach 68·5, the steam is turned off.   It is safe to assume that by the time the jam has cooled in the jars, the solids will have risen to 69·5 per cent.   When the steam is turned off, the jam is quickly tipped out into a transport bogie which can be wheeled to the weighing machine.   If the pH requires adjustment, powdered citric acid can be added either at the beginning or the end of the boil, according to whether the invert is above or below the limits.   High acidity can also be corrected by the addition of sodium bicarbonate but care should be taken not to use this substance in excess as it spoils the colour of the jam.   Usually 1 to 3 oz. of citric acid per 100-lb. boil is all that is required while the use of more than 1 oz. of bicarbonate should be avoided.   Sodium citrate can also be used to reduce the acidity.

Where vacuum pans are used, sugar, water and pectin are heated together to about 140° F. in open pans.   The fruit is then added and several batches of this mixture are transferred by suction to a large vacuum pan where boiling is finished, under a vacuum of 25 to 28 inches at a final temperature of 140° F.   The preserve is then pumped to the filling lines.

FINISHING AND STORING

Inversion of sugar is greatly affected by temperature.   It is therefore important in the control of this factor that the jam should be cooled as quickly as possible after boiling.   Also prolonged heating tends to caramelise the jam and while it remains hot, difficulties are encountered, particularly in whole fruit strawberry and marmalade with the fruit or peel floating to the top of the jars.   The jam as it comes from the boiling pans will be at a temperature of over 212° F. and it must be cooled to 185° F. to 190° F.   This can be accomplished in tanks with double walls through which cold water is flowing or in a revolving cylinder surrounded by a water jacket.
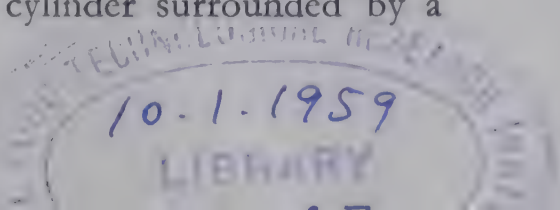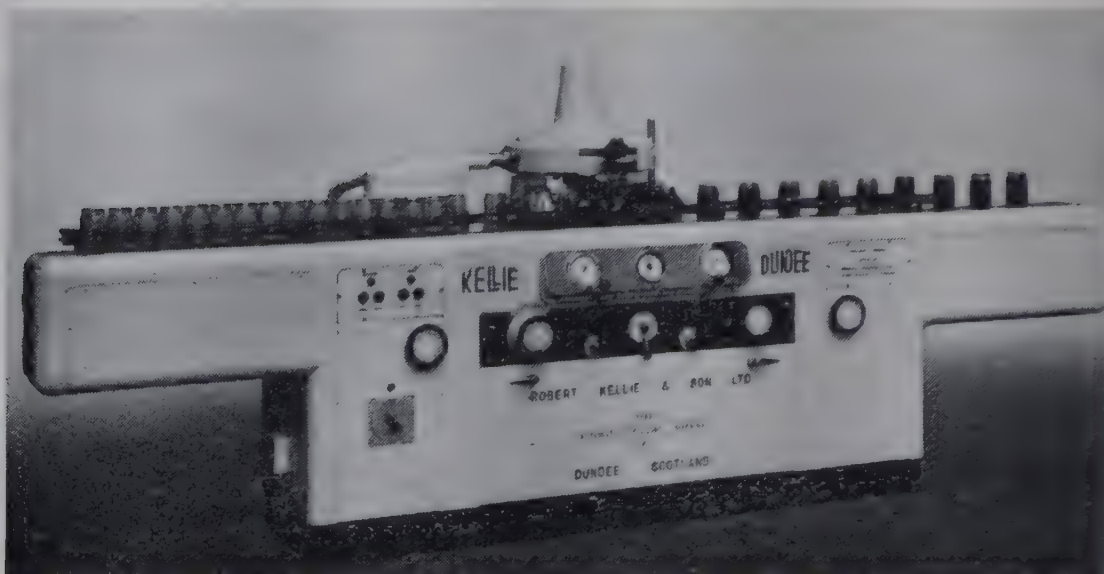
FIG. 4.—JAM FILLER.

Empty jars are placed on one side of this machine and full one removed from the other side.   All controls of amount of fill, speed of machine and filling temperatures are available on the front of the machine.

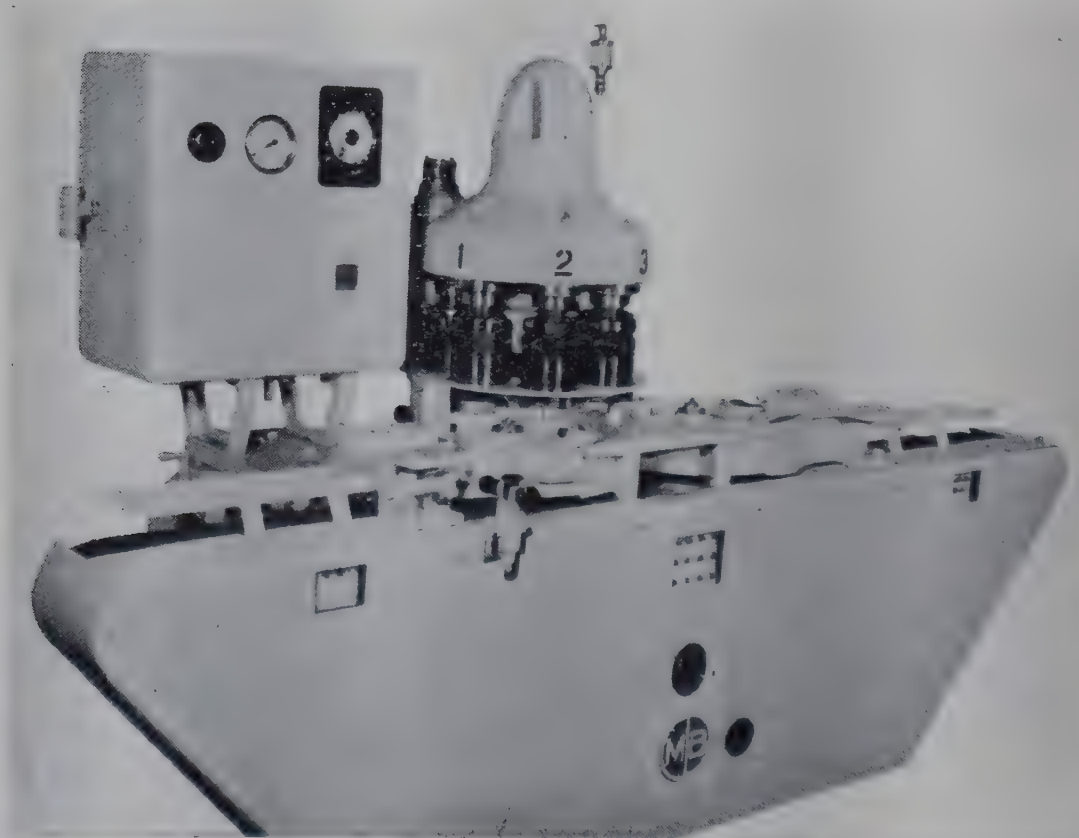(*Robert Kellie & Son, Ltd.*)

FIG. 5.—MACHINE FOR APPLYING VACUUM CAPS.

This machine known as the "Garda" capper applies one-piece aluminium caps with a plastic gasket.   Steam is injected into the headspace before the cap is finally sealed.

(*The Metal Box Co., Ltd.*)

Filling Containers By Hand

The cooled jam can be filled into containers either by hand or machine. Hand filling is slow but is preferable where only small lots are being made or large containers, such as 28 lb. tins, are required. It also avoids the breaking up of whole fruit which might occur in a machine. The usual method of hand filling is on circular rotating tables. The jam is brought to the tables in bogies and poured into the jars, which are arranged around the edge of the table, by means of a dipper which has a funnel shaped end. After filling, the operator makes the table rotate so that the filled jars pass round to another worker. A disc of tissue is then laid flat on the surface and the jars removed to a truck on which they can be transported to a cooling room.

Filling Machines

For a high rate of production a filling machine is indispensable. The jam is fed from the cooler into a sump from which it is drawn by means of a double acting plunger-type pump which in turn delivers it in fixed amounts to the filling nozzle of a rotating head. The exact amount delivered can be adjusted by means of a hand wheel which regulates the length of the stroke of the pump. The empty jars are placed on shoes attached to one side
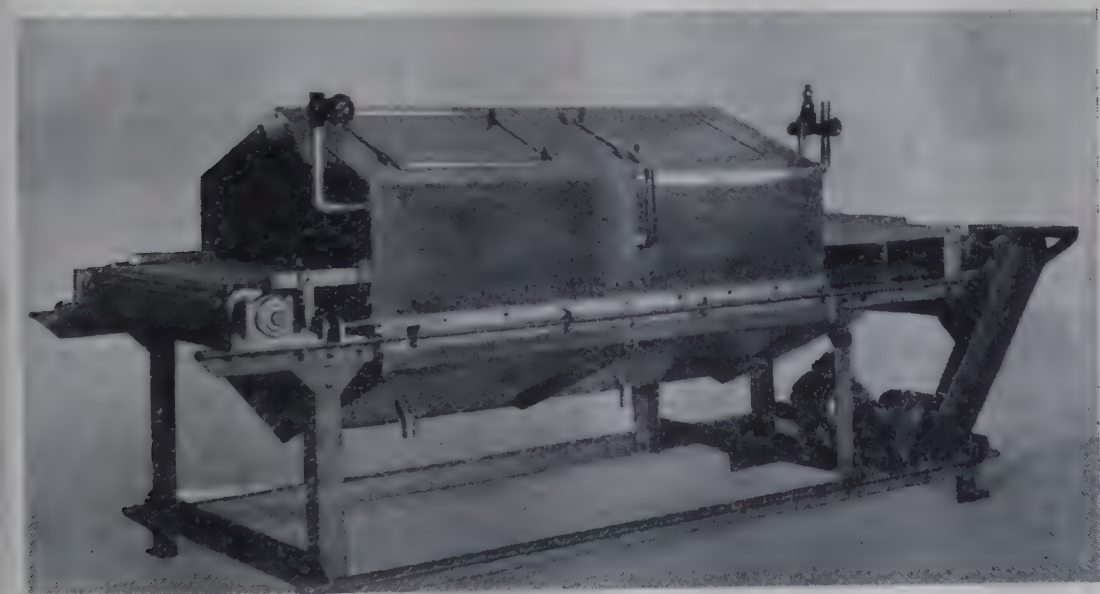
FIG. 6.—JAM STERILISER.

Vacuum packed jars should be sterilised by steaming for three minutes in a broad belt steam tunnel such as illustrated.

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of an endless chain. They pass under the nozzles of the filling head and round to the delivery side when tissues are placed on the surface of the jam. The filled jars are removed from the shoes and stacked on trolleys for removal to cooling rooms.

Sealing Methods

The closing of containers of jam can be done in a variety of ways. Open top cans are closed by seaming on the lid with a double seaming machine and then passing the cans, without agitation to break the set, through a water bath. Jars can be closed with clip-on lids or by some form of seamed-on lid. If the jar is hermetically sealed then it is advisable to pass it through a steam chamber to sterilise the surface and the lid. In some plants a type of closure, fitted with a rubber gasket, is used which seals on to the rim of the jar as it cools making a vacuum closure. Another type of capper contains an arrangement whereby a jet of steam fills the headspace just before the cap is sealed on to the jar.

The advantages of vacuum packing of jam are that an hermetic seal is formed under conditions which discourage mould growth. The use of paper tissue is eliminated and the jars can be mechanically washed. They can then be stored indefinitely, even if the solids content is as low as 65 per cent, without incurring mould growth. Where a clip on lid is used the soluble solids should not be less than 69·5 per cent. The presence of the disc of tissue is intended to prevent the condensation of moisture on the surface of the jam. This condensation would create localised areas of low concentration in which mould would readily grow.

J. H. C.

CHAPTER 5

EDIBLE FATS—SHORTENINGS

SHORTENING is a collective term applied in food technology to describe lard and lard substitutes used in the baking of bread and the production of cakes, pastry and biscuits. Such fats are used either to form or to increase the fat-content of the product, or to induce crispness or flakiness, particularly in pastry and biscuits. Just as butter derived from milk was, until the introduction of margarine, the fat for direct human consumption, so suet and lard were until comparatively recently the only practicable form of shortenings. Today, however, natural lard and suet are being increasingly supplemented by various prepared fats for shortening purposes. Known as lard compounds or lard substitutes, most of these substitutes for natural shortenings are derived from vegetable sources.

MANUFACTURE OF LARD

By far the greatest proportion of natural lard comes from the bodies of pigs, cattle and sheep, in that order. The main centres of production are the United States of America, followed by Germany and Denmark. Every year these countries produce approximately 1,000,000 tons of natural lard, of which about 100,000 tons are imported by the United Kingdom. Lards are usually graded for quality according to the standards obtaining in the Middle West of the United States. The average analytical characteristics of lards used as shortenings are as follows:

Specific gravity at 15° C.	0·934–0·938
Melting point	28°–48° C.
Setting point	22°–32° C.
Acid value	0·5–1·3
Iodine value	46–66
Ref. index.	1·441/60°

Best quality lard comes from the fat surrounding the stomach and kidneys of the pig; although, as already stated, sheep and

cattle are also valuable sources. As soon as possible after the animals have been slaughtered, the lard-yielding parts of the carcases are scraped to remove adhering non-fatty tissue, followed by thorough washing in water. The parts are then sent to chopping and mincing machines which reduce them to pieces sufficiently small for rendering. Rendering can be by either the dry of wet process.

Dry Rendering

Dry rendering vessels are made of iron, either enamelled inside or lined with tin, the latter being the more common. Where the rendering process is to be carried out at temperatures below 100° C., the vessels consist of large-capacity rectangular or cylindrical tanks fitted with closed and open steam coils and provided with a light lid to prevent the entry of dust. The tanks have mechanical agitators to keep the contents in constant movement and are fitted with skimmer pipes through which the liquid fat is removed from the surface of the contents when rendering is complete. The temperature inside the tank is maintained at between 40° C. and 50° C., and in large plants is thermostatically controlled. As the temperature rises within the vessel, the fat gradually escapes from the cell tissues and floats to the top, while the tissue, which still holds appreciable quantities of fat, sinks to the bottom of the vessel.

Wet Rendering

There are two methods for rendering fat in the presence of water; that is, the non-dry system. One is in open digesters, where the temperature does not exceed 100° C.; the other is by autoclave at temperatures in excess of 100° C.

In the open digester, fresh material, or the fat-bearing-tissue residue from the dry-rendering process, is placed in tanks containing water in equal amount to that of the fatty material. The contents are then brought to a temperature of not more than 100° C. either by steam in closed pipes or by the open admission of steam. Rendering at that temperature in the presence of water, yields the highest quality lard. Increasing the rendering temperature up to that of boiling water yields a lard having a high fat content, but of somewhat lower quality. As in the dry process, the fat rises to the top of the vessel, and the separated

residues fall to the bottom. These residues hold an extractable quantity of fat which can be obtained by rendering in an autoclave.

Rendering autoclaves consist of tin-lined, jacketed vessels having open steam coils and able to work up to a maximum pressure of 150 lb. per sq. in. The fat charge, which may consist of either the residue from an open digester or new fatty material, is laid on a perforated false bottom in the autoclave and a small amount of water is added. The temperature inside the autoclave is then raised by a current of open steam. The process continues for several hours, usually at a pressure of about 85 lb. to the sq. in. This causes the tissue to surrender practically all its fat content, which is drawn off after the pressure has been released inside the autoclave and the fat and residue have settled.

Lard obtained by the intensive rendering in autoclaves is not of such high quality as that yielded by low-temperature rendering processes. This is because non-fatty organic matter is more liable to be separated at high temperatures and to pass into solution in the fat. Moreover, high temperatures are liable to induce hydrolytic action, resulting in appreciable production of free acidity in the fat.

Qualities of Lard

Dry and wet rendering yield four qualities of lard; neutral lard, No. 1; neutral lard, No. 2; leaf lard; and prime steam lard. The first is obtained by rendering kidney and bowel fat with or without water at temperatures below 50° C., while the second is derived from back fat, similarly rendered. Leaf lard is obtained from the residues of Nos. 1 and 2 neutral lards rendered in autoclaves, and prime steam lard is derived from the autoclave rendering of any fatty parts of the carcase except the back, kidneys and other intestinal organs.

From these various qualities of lard, which vary in composition according to the parts of the animal from which they are obtained, and in consistency are governed by the rendering methods employed, lard oil and lard stearin are prepared by a moderate chilling process followed by pressing. All these qualities of edible lard are suitable for use as shortenings, but the choice of any particular quality must be governed by the quality aimed at in the production of the bread, pastry or other cooked product. An efficient shortening, whatever its purpose, should be of a

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soft, granular texture, and fairly firm without being brittle or over-cohesive.

Suet is often retailed as a shortening for domestic cooking. Suet of this nature has not been rendered, but is taken direct from the carcase and cleaned. Consequently, it contains a certain amount of tissue or "skin." There are also a number of proprietory suet shortenings, retailed for domestic use, in which the suet has been cold rendered in factories to remove the tissue. Premier jus is suet that has been rendered without water at temperatures below 50° C., while oleostearin is derived from premier jus under pressure. Neither premier jus nor oleostearin are much used as shortenings, their principal applications being in the manufacture of margarines.

LARD COMPOUNDS

Lard possesses the qualities desired in a commercial shortening to a greater extent than in any other substance, and, indeed, than in any other animal fat. Nevertheless, commercial demand far exceeds the supply, while the cost of animal lard has risen steadily during the past 40 years. Consequently, scarcity and economic necessity have brought into use various blended mixtures which in practical applications closely simulate the best animal lard. These mixtures, which are now extensively used as commercial shortenings, form the group called lard substitutes or lard compounds.

Manufacturers of lard substitutes have been remarkably successful in producing a material that very nearly approximates animal lard in texture and physical consistency. The best of them induce in the baking mixtures in which they are incorporated the shortening or crispening effect produced by good quality animal lard, and they are now extensively used in commercial practice.

Lard substitutes, or, as they should be more correctly called, lard compounds, were first made by thoroughly mixing premier jus or oleostearin with a liquid vegetable oil such as that derived from maize or cottonseed. The mixture is melted and then rapidly cooled. Cooling is carried out by feeding the mixture into a trough in which revolves a narrow cylinder warmed internally by water at a temperature of approximately 35° C. A second cylinder, but of larger diameter, and cooled internally by a refrigerant solution of calcium chloride at a temperature of

approximately —7° C., is mounted in the trough in such a position that it is in contact with the warm and smaller cylinder and rotates in the same direction.

Suitable adjustment of the rates of rotation of both drums causes the smaller drum to pick up the liquid emulsion and throw it on to the larger cooling drum. There it is retained for a considerable proportion of a revolution until it is cooled and so solidified. A fixed horizontal scraper then removes the mixture, which falls into a collecting trough. There a rotating conveyor working on the principal of the Archimedean screw pushes the chilled emulsion forward to a rolling machine consisting of large revolving drums fitted with internal rollers. The rolling machine kneads and presses the solidified mixture into a coherent mass from which all but some 13–16 per cent of the water content has been forced out. A final process further beats up the solidified mixture until it becomes a fine, granular, opaque mass which closely resembles lard in appearance.

Simple lard compounds of the type described above now have to compete with substitutes made by the partial hydrogenation of liquid fats obtained from cottonseed, maize, sunflower, soya, groundnut, or whale oil. Cottonseed oil is the one most commonly used in the United States, and is there considered the most efficient, but in Britain the preference is for soya and groundnut oils.

HYDROGENATED OILS

Cottonseed oil is present to the extent of about 40 per cent in the seed which is left after the removal of the raw cotton fibre. It is produced in large quantities in the United States of America and in India, and in Europe from imported Indian and Egyptian seeds. Maize oil, or, as it is sometimes called, corn oil, is produced in large quantities in the United States by expressing it from the seeds of Indian corn. The seeds have an oil content of about 50 per cent. Sunflower oil is obtained from the seeds of the common sunflower, the seeds consisting of about 30 per cent oil. It is more popular as a constituent of lard substitutes on the Continent than in Great Britain.

Whale oil is derived from the blubber and flesh of a number of different species of whale, ranging from the "Right" or Greenland whales of the North Atlantic to the South Sea whales of the South Atlantic and Pacific oceans. The oil is extracted

and refined on the factory ships accompanying the whaling fleets, the best quality being grade O, which is colourless and lacks free fatty acid. The quantities of whale oil produced annually are limited by an international agreement designed to prevent complete extinction of the whales by over-catching. About 90 per cent of the whale oil landed in the United Kingdom is for the production of lard compounds and margarine.

Preliminary Refining

For the production of lard compounds used as commercial shortenings one or other of the foregoing oils undergo a preliminary refining by an alkali process. Since the oils are required in a solid or semi-solid condition they are hardened by a process of hydrogenation. This consists in saturating the unsaturated fatty acids by the addition of hydrogen at the double bond, e.g.:

$$- CH = CH - + H_2 \longrightarrow -CH_2 - CH_2 -$$
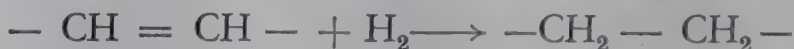
As hydrogen enters the oil molecule, the fall in iodine value is accompanied by a rise in melting point. Thus, a fully hydrogenated whale oil, which originally melted at below 0° C. has a melting point of about 55° C. Certain of the vegetable oils with natural melting points in the region of 3° C. have, after hydrogenation, melting points of the order of 69° C. to 70° C.

Hydrogenation

Hydrogenation is effected by reacting the well-purified oil with hydrogen in the presence of finely-divided nickel to act as a catalyst. The reaction occurs in vessels surrounded by a heating jacket and fitted with a mechanical agitator of the cyclone type. Hydrogen is passed up through the heated oil, in which the nickel catalyst is suspended, and out through the top of the vessel. Throughout the process, the internal temperature of the vessel is kept at 180° C. and an internal pressure of about five atmospheres is maintained. In another process, called continuous hydrogenation, the oil is caused to trickle down over the nickel catalyst, which is in the form of turnings or a wool, while hydrogen is passed through the long, narrow, vertical vessel in which the operation takes place.

Deodorisation

After hydrogenation, the oil goes through another refining process and is then deodorised. Deodorisation is necessary to

remove from the oil ethereal, hydrocarbon, or other substances which, even when present in only small quantities, often possess distinctive taste and smell. Hence they must be completely removed before the fat is suitable for use as a shortening.

Deodorisation consists of heating the fat in closed aluminium or iron vessels to a temperature which may vary between 160° C. and 220° C. The heating is by high-pressure super-heated steam at 200° C. passing through a coil at the base of the vessel. The vessel itself must be exhausted to the highest practicable vacuum. The heating process lasts from 10 to 12 hours according to the kind of oil, and at the end of that period the oil is cooled down to a temperature of below 100° C. It is then ready for use.

Vitamin Addition

Unfortunately, the high temperatures essential to effective deodorisation inevitably destroy any vitamin content of the original oil, and this defect could be overcome only by some deodorisation process employing a low temperature. At present, the problem of devising such a process seems insoluble. For that reason, and that reason only, lard substitutes are somewhat inferior to animal lards; although the vitamin content of a shortening is not particularly important, as shortening is not primarily intended to increase the nutritional value of a baking or other mix.

Nevertheless, the industries concerned with the production of commercial shortenings have devoted considerable research towards efforts to renew the vitamin content lost by deodorisation of lard substitutes. Already a certain measure of success has been achieved, and it is now possible to induce vitamin D in certain of the deodorised oils by ultra-violet irradiation. On the other hand, vitamin A cannot be introduced in the same way. It is, however, possible to obtain concentrates of both vitamins A and D from suitable sources and to add the necessary proportions of these to the deodorised oils. This is now the common practice with manufacturers of lard substitutes used as commercial shortenings.

Cooling

To return to the actual production of lard substitutes from vegetable oils—After completion of the deodorisation process,

the lard substitute is cooled and then chilled in a trough by means of a chilling drum like that used in the manufacture of the animal and vegetable lard compounds. The resultant product constitutes a shortening in every way as suitable as the beef-stearin-cottonseed-oil types. Moreover, the texture approximates fairly closely to that of good quality animal lard.

COMPOUND SHORTENINGS

Hydrogenated fat having the requisite iodine value is often modified for shortenings by mixing with a comparatively hard hydrogenated product, having a melting point in the region of 50° C., some refined and deodorised but unhydrogenated oil. Alternatively, two qualities of hydrogenated oil may be mixed together, such as a hard product with a melting point of about 40° C. and a softer product with a melting point of approximately 20° C. Practical tests in bakeries have proved that the consistency of hydrogenated fats so blended is more suitable as shortening for biscuits and pastry than is a straight hydrogenated vegetable-oil shortening.

Another advantage of using a shortening compounded from hard hydrogenated fat and the original unhydrogenated oil is that the shortening is less liable to induce "hardening flavour" in the baked product. Shortenings of this nature have, however, the serious disadvantage that the linoleic glycerides in the unhydrogenated portion of the mixture induce a tendency to rancidity. Experience indicates that for the baking of pastry or crisp biscuits the best results are obtained by using a shortening having a melting-point in the region of 40° C. Whether or not a blend of hard and soft hydrogenated fats, a shortening with a melting point of that order is less liable to induce off-flavours.

STABILITY OF SHORTENINGS

Because fatty matter is an ideal breeding-ground for many forms of mould and bacteria, the preservation of shortenings in a fresh state is a matter of the utmost importance. When they are no longer fresh, shortenings, like all fats and edible oils, become rancid and, therefore, contaminate the biscuit, pastry or other mix in which they are incorporated.

As yet, the chemistry of rancidity is somewhat obscure, but it

is known that the action of ordinary atmosphere is a predisposing cause. Although of widely differing chemical composition, all fats and oils contain glycerides of the fatty acids. When fats and oils are exposed to light and air, the glycerides decompose, so increasing the acidity that induces formation of free fatty acids. It is the characteristic taste and smell of these free acids that betray rancidity.

Whilst a definitely rancid shortening immediately betrays itself by taste and odour, incipient rancidity is not always easy to detect. There are, however, a number of comparatively simple chemical tests which immediately reveal incipient rancidity; these include the Kerr or Kreis test and the Issoglio test (*see* p. 207).

Both these tests indicate the presence of rancidity, but it is obviously more important to be able to assess the liability of a shortening to develop rancidity. This can be done by the peroxide value test (*see* p. 206) so called because organic peroxides are among the early products of the atmospheric oxidation of unsaturated fats that induce rancidity.

Careful storage of shortenings is essential to prevent the development of rancidity, and particular care must be taken in the type of vessel in which shortenings are kept. Many metals and their salts are exceptionally efficient catalysts for the oxidation that induces rancidity. Nickel, copper, vanadium, cobalt and iron, or compounds of any of these, must be avoided. The best storage vessels are of earthenware, or if metal must be used, of chromium-nickel steel, aluminium or tin. Above all, when it is necessary to store shortenings for any length of time, they must be kept absolutely sterile, excluded from contact with the atmosphere, and not be exposed to contamination by bacteria or other harmful organisms.

Atmospheric oxidation of fat is accelerated by the action of light, particularly by ultra-violet rays within the wave band range of 290 to 400 m.μ. and also by visible rays in the blue region of the spectrum below the wave band range of 500 m.μ. Conversely, any light with a wavelength exceeding 500 m.μ. has comparatively little effect on rancidity. Hence it has been suggested that stored shortenings should be protected by wrappers of a colour that will not admit light of the rancidity-activating wavelength.

D. Le R.

MARGARINE

THE origin of margarine and the success which it has attained as an established part of our diet can reasonably be attributed to the succession of wars in Europe.

Margarine was invented at the time of the Franco-German war as a substitute for butter and has always been regarded as such, although of latter years much of the popular prejudice has been overcome.

The principles underlying margarine manufacture have not materially changed over the years but the procedure has become modernised, largely by a change from batch processes to continuous methods.

The Three Main Types

Margarine is essentially an emulsion of soured milk and brine with oils and fats, but if an unsalted margarine is required it is usual to omit the milk as well as the brine and use only water in their place. In the main there are three types being manufactured today: (1) Domestic or table margarine for home use; (2) cake margarine for the bakery trade and for other manufacturing purposes, while the ice-cream manufacturer often uses unsalted cake margarine; (3) pastry margarine; again for the baker, but as the name implies it is specially for making pastry.

INGREDIENTS

The butter-like flavour of margarine is controlled to a considerable extent by the selection of the milk and its treatment.

Milk

Good quality skim milk or reconstituted powder milk is pasteurised and after cooling is soured by inoculating with a culture prepared from specially selected lactic acid bacteria. The proper control of this souring process is of extreme importance as a variation in temperature can materially affect the aroma of the soured milk and in turn the flavour of the finished margarine.

Whereas at one time the culture used would only contain strepto-coccus lactis it is now recognised that the cultures giving the best aroma will always contain two or more different organisms and those commonly used are *S. paracitrovorous*, *S. citrovorous* and *S. cremoris*.

It is usual for the souring to take place overnight, taking about 16 to 18 hours, with the temperature controlled at 20° C. to 22° C. The correct degree of souring is reached when the pH is 4·5, the milk should then be cooled as low as possible to prevent the further development of acidity. It has been found that the aroma produced is due to the development of diacetyl and acetyl methyl carbinol and consequently these substances are often used as the basis of synthetic butter flavours which are sometimes used to assist in the final flavour of the finished product. Although margarine can be made without souring the milk, it is not regarded as a desirable practice as by so doing there is no control of the aroma and many undesirable flavours may be introduced due to the development of foreign bacteria.

Oils and Fats

The next stage is the selection of the oils and fats and mixing them in the correct proportion in preparing the fat blend. The selection and proportioning is of great importance as it controls the consistency of the finished product. One might say that any fat which is of satisfactory physiological standards can be used but those in universal use which are solid at ordinary temperatures are probably coconut oil, palm kernel oil and palm oil. Of those liquid at ordinary temperatures, ground nut, cottonseed, sun-flower and soya bean oils are most commonly used.

To the foregoing must be added the important and ever-useful hydrogenated fat. Hydrogenation enables whale oil to be used, a fact of great economic importance. The melting point can be raised to above 50° C. and at the same time all trace of fishiness is removed.

The most usual melting point to be used for margarine manufacture is 48° C. or 42° C., sometimes a lower melting point is used, i.e., 32° C., but this is not so stable and there is more risk of reversion of flavour. It can be said, that today hydro-genated whale oil has replaced the premier jus which was used in making margarine during the 1914–18 war. Other oils can be

...ccessfully hydrogenated for use in making margarine especially groundnut, cottonseed and palm oils.

Formulation

When computing the formula, attention must be given to the purpose for which the margarine is required. A table margarine must be plastic at ordinary temperatures, so as to spread freely, this is brought about by varying the proportion of hard and soft oils according to the season of the year. This gives margarine a great advantage over butter which is not easily spreadable in the winter and is often too soft in the summer.

As a guide as to what might be considered a reasonable formula for a table margarine in the summer, one could use 23 per cent hardened whale oil 46° C. to 48° C., 12 per cent hardened groundnut oil 33° C. to 35° C., 43 per cent coconut or palm kernel oil, 8 per cent palm oil and 14 per cent, groundnut or other liquid oil. A cake margarine can be made of approximately 25 per cent hardened whale oil 46° C. to 48° C., 60 per cent coconut or palm kernel oil and 15 per cent liquid oil.

A pastry margarine must have a very tough consistency but at the same time be pliable, it is worked into the dough in layers (alternate dough and fat) by repeated rolling and folding over. On baking this produces what is known as puff pastry. A reasonable formula for this type of margarine could be 43 per cent hardened whale oil 46° C. to 48° C., 15 per cent beef stearin and 42 per cent liquid oil.

Oils and fats used for margarine should be of the highest quality, they should be odourless, tasteless and colourless. The free fatty acids should be less than 0·1 per cent and the peroxide value of the mixed blend should preferably not be above 1·0. Animal fats do not play an important role in margarine manufacture today, in fact their use has almost ceased except for some pastry margarines. An important factor in the final selection of any oil or fat is its current market price.

BATCH MANUFACTURE

The principal ingredients have now been considered and there remains the salt which at one time was added in the dry state when kneading but is now invariably dissolved in water, making a strong brine, which is introduced with the milk.

The next stage is the emulsification of the fat blend with the milk and brine. This can be done in batches, usually about 1 ton, or by means of continuous emulsifying pumps. This latter system is gaining popularity because of the saving of space, equipment and labour, although there is a section of opinion against it as often a very fine emulsion is formed which tends to give the margarine a greasy appearance.

Emulsification

In the batch system the fat blend and aqueous phase (milk and brine) are run into an oval-shaped vessel known as a churn which is fitted with two gate stirrers which revolve at a speed which can be varied from 20 to 90 r.p.m. The whole is emulsified by agitating at top speed while diminishing the temperature, the operation taking about 20 to 30 minutes, when the emulsification is complete the emulsion is run slowly on to a revolving cooling drum which is refrigerated by the direct expansion of ammonia.

Cooling

On coming into contact with the drum the emulsion sets hard and forms a thin film which remains on the drum for one revolution when it is scraped off by means of a knife into thin flakes. Cooling drums vary in diameter from about three feet to seven feet and consequently the speed of rotation and its temperature will vary as well as the thickness of the film. However, it is usual for the thickness of the film to be in the region of four- to eight-thousandths of an inch, and the temperature of the flakes after leaving the drum to be a few degrees above O° C.

Kneading

After the flakes leave the cooling drum there are two different methods of treatment. The older method is to pass the flakes through a series of three or four pairs of granite rollers known as a multiplex. These rollers knead the flakes together and then pass the mass on to a circular revolving table with a paddle fixed from the centre to a position on the circumference. The table and the paddle revolve and so the margarine is worked and kneaded to give it the desired texture.

Blending

When this operation is complete the margarine is finally worked in a blending machine fitted with two S-shaped paddles

revolving at high and different speeds and is similar to the dough kneader used by the baker. It is here that the moisture content if too low, can be adjusted. The modern type of blender is made to operate under vacuum thus avoiding the incorporation of excess air and so reducing the consequent risk of oxidation.

The more modern method of treating the flakes after leaving the cooling drum and the one which requires much less labour and equipment is to pass the flakes through a continuous kneading machine of the type often known as a complector and which is of Scandinavian origin. With this system the flakes are pressed and passed between two rollers and then forced under vacuum through a series of perforated plates when it emerges completely plasticised and ready for packing.

CONTINUOUS PROCESS

In contrast to the methods just described there are some continuous systems in operation for the manufacture of margarine and although their use is gradually gaining ground there is still a strong body of opinion that the texture of such margarine is not as good as that produced by the older methods. Probably the best-known continuous method is that known as the Votator system. In this the aqueous phase and fat blend are mixed and then passed through a reciprocating pump to an enclosed chilling unit which again is refrigerated by the direct expansion of ammonia.

This unit consists of one or more cylinders containing a revolving mutator shaft on to which is fitted scraper blades which remove the margarine film from the surface immediately it is formed and at the same time causes some agitation as the margarine is passing on to the next unit. In this unit the solidified mass comes to atmospheric pressure and passes through without any further working, although sometimes a screen is fitted for further texturating, when it is ready for packing. This is done by automatic machinery which packs at speeds varying from 60 to 120 half-pound packets a minute.

ADDITIONAL INGREDIENTS

Margarine is made to resemble butter in appearance by the use of colouring materials which are added at the emulsification stage. Coal tar dyes, annatto or carotene can be used and

although some parts of the country have a preference for a lighter and some for a darker margarine, it can be stated that the intensity of the colour does not bear any relation to the flavour of the margarine. When carotene is used for colouring it also contributes a part of the Vitamin A content.

Vitamins

It is now illegal to manufacture table margarine without the addition of vitamins and the required potency is 760 to 940 I.U. Vitamin A, per ounce (27 to 33 per gram), and 80 to 100 I.U. Vitamin D, per ounce (approximately 3 per gram), which in this respect makes margarine equal to if not superior to some butters. The vitamin concentrate is added at the emulsification stage and the Vitamin A being unstable immediately loses part of its potency so that it is necessary to add about 10 to 15 per cent in excess of the theoretical requirements in order to maintain the above standard.

Emulsifying Agents

Often special agents are added to the fat blend to assist in forming and maintaining the emulsion. Margarine made by the churning method without the use of an emulsifying agent is liable to be loosely bound and will then show a tendency to exude the aqueous phase after packing thereby reducing the keeping properties.

Probably the earliest emulsifier to be used was egg yolk. Later commercial lecithin was introduced. It is still used by some manufacturers because (in addition to its emulsifying properties) it is reputed to act as an anti-oxidant. Moreover, when used in frying, it has the effect of producing browning similar to that produced by butter. Oxidised and polymerised soya bean oil has been used but the modern practice is to use mono- and diglycerides which are very effective in small quantities, requiring only about one or two parts per 1,000 of fat blend.

LEGAL REQUIREMENTS

There is no legal limit to the amount of salt which margarine may contain, the usual range is 1·5 to 2·0 per cent. Margarine, like butter, must not contain more than 16 per cent of water. There is a stipulation that butter cannot be added to margarine in excess of 10 per cent. The use of preservatives is not allowed,

although during the war years it was permissable to use borax and boric acid up to 0·25 per cent calculated as boric acid.

It is preferable not to keep margarine in a refrigerator (as this is detrimental to the texture), but rather to store it in a cool place at about 45° F. to 50° F. and to consume it within three weeks of manufacture.

PLANT—MATERIALS OF CONSTRUCTION

Today, stainless steel probably predominates, as it can be used for any of the plant required, for the manufacture of margarine. It is particularly useful for the dairy equipment and it has a first-class appearance. Aluminium has been used for the dairy equipment and for the trucks used for transporting the margarine from one part of the factory to another, however, this is now being superseded by stainless steel. Iron and copper, if they cannot be avoided, must be well tinned and the tinning watched for wear as these metals rapidly promote oxidation.

Cleanliness of the plant and factory is extremely important and should be watched by the bacteriologist to check the development of undesirable bacteria, moulds and yeasts which would cause a diminution in the keeping qualities of the margarine.

DIETETIC VALUE

It is generally agreed that the calorific value of margarine is equal to that of butter and that its digestibility is also equal to butter.

Mention has already been made of the addition of vitamins to domestic margarine. This addition was in practice on a voluntary basis for several years before the last war, but is now compulsory; thus margarine often has a higher vitamin content than butter, especially that produced during the winter months.

The description of margarine may be closed with an extract from a report of the Food and Nutrition Board of the National Research Council of America:

The present available scientific evidence indicated that when fortified margarine is used in place of butter as a source of fat in a mixed diet, no nutritional differences can be observed. Although important differences can be demonstrated between different fats in special experimental diets, these differences are unimportant when a customary mixed diet is used.

G. H. C.

FLOUR MILLING

THE conversion of wheat into a *meal* for the production of baked goods for human consumption is an ancient craft but the basic principle of the present-day system of milling wheat into *flour* was introduced less than 75 years ago. The difference between the old and the new methods is great; a meal is produced by pulverising the grain, whereas the production of a flour involves dissection of the kernels, whereby the inner starchy matter is separated from the outer skins. A proper understanding of modern milling methods, which have been justly referred to as "biological engineering," therefore calls for a knowledge of the structure of the wheat grain.

THE STRUCTURE OF THE WHEAT GRAIN

A wheat grain consists broadly of three main fractions, the germ, the endosperm, and the outer skins. The disposition of these fractions is shown in the longitudinal section of a wheat kernel depicted in Fig. 1.

The germ is the vital part of the grain which gives rise to the new plant when the grain is submitted to conditions favourable to growth. It represents about 2·5 per cent of the grain and consists of the embryo proper and the scutellum, which is a membrane separating the embryo from the endosperm. The function of the endosperm is to serve as a source of food for the young plant until such time as the root system is sufficiently developed to withdraw the required nutrients from the soil. The endosperm content of the grain is about 83 per cent. The skins serve as a protective envelope for the endosperm when the grain is sown as seed. They account for about 8 per cent of the grain. Interposed between the endosperm proper and the outer skins is the aleurone layer which is a specialised part of the endosperm, although it is normally removed with the skins during milling. It represents about 6·5 per cent of the grain.

BEARD

SKINS

ENDOSPERM

GERM
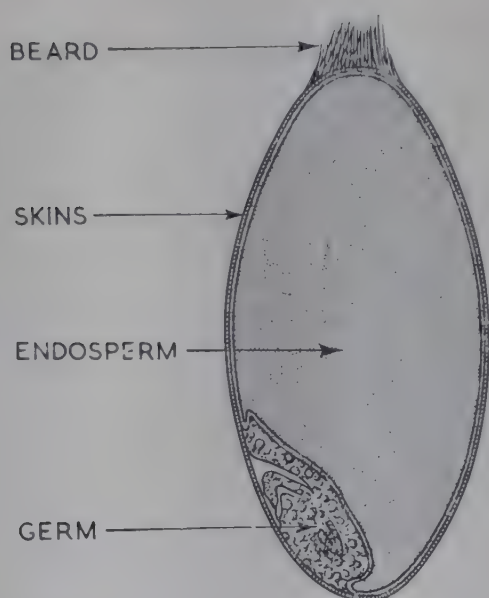
FIG. 1.—LONGITUDINAL SECTION OF
WHEAT GRAIN.

The aim of a miller who is making white flour is to split open the grains of wheat and to scrape out some of the endosperm in such a way that it is contaminated as little as possible with particles of skin. In doing this, he endeavours at the same time to separate as much of the germ as he can. The amount of endosperm which a miller removes as flour, expressed as a percentage of the weight of the wheat used, is referred to as the extraction; a flour of 72 per cent extraction, for example, would be arrived at by finishing the milling operation with 72 parts of flour and 28 parts of by-products for every 100 parts of wheat sent to the mill.

It is not possible to remove from the grain a reasonable proportion of the endosperm completely uncontaminated with ground-up skins, that is, free from "bran powder," but the lower the extraction the lower the bran powder content. Conversely, it is not possible to scrape the skins entirely free from endosperm, and so the by-product, bran, which is the opened-up and flattened skins of the grains, always contains adhering endosperm. Toward the end of the long series of operations that constitute the flour milling process a material is obtained which consists of an intimate mixture of endosperm and finely ground-up skins from which it is impossible to separate the endosperm. This by-product of flour milling is known as Weatings, sharps or middlings.

THE MILLING PROCESS

Before the application of the operations which convert wheat into flour, the grain is subjected to a very thorough cleaning. It is passed over sieving machines designed to remove impurities which are smaller and others which are larger than the grain, and during the sieving processes aspiration is applied whereby light pieces of skin which may have become detached from the surfaces of the grain, light seeds and dust are removed. The grain is
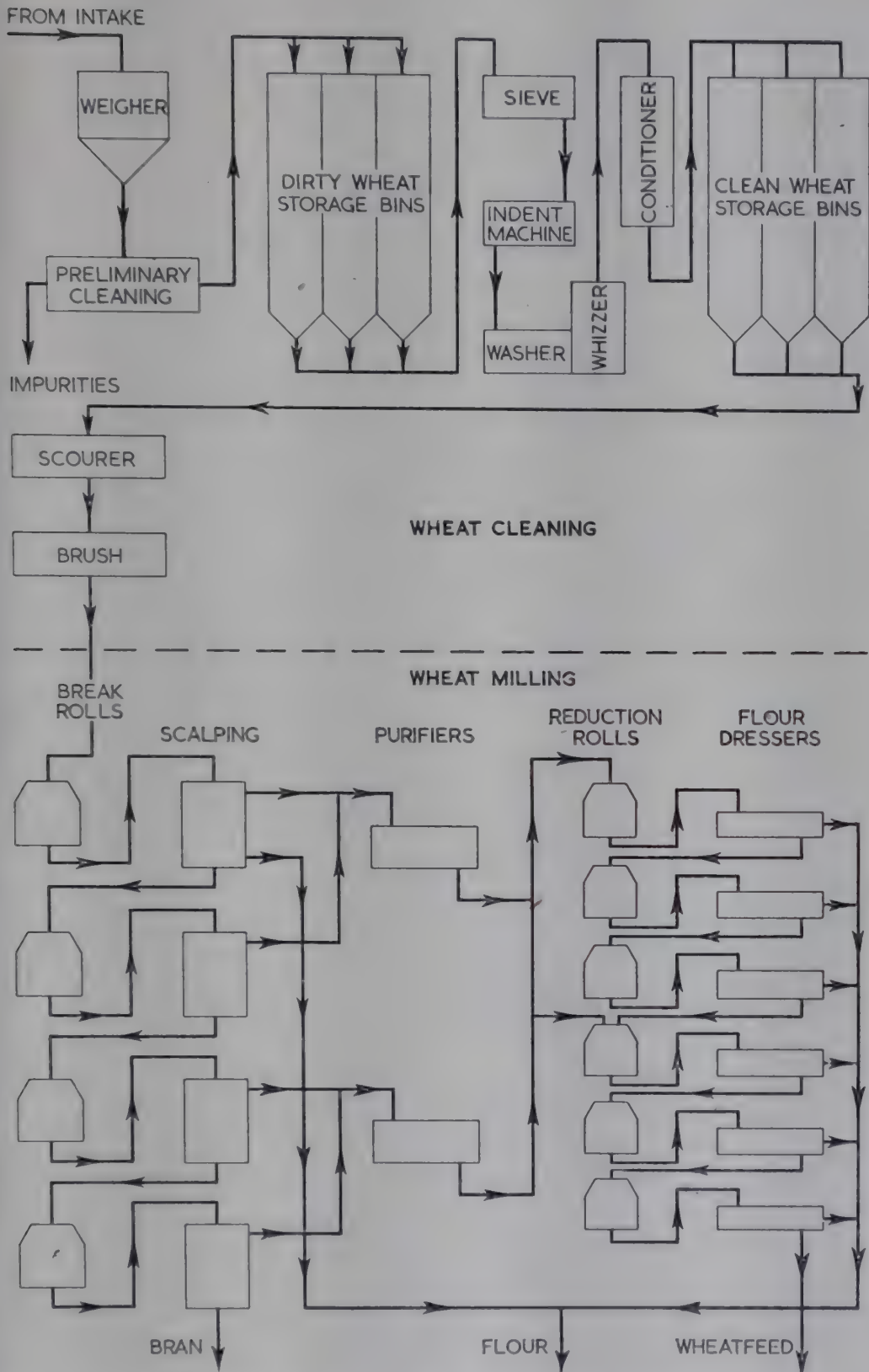
FIG. 2.—SIMPLE DIAGRAM OF BASIC OPERATIONS OF WHEAT CLEANING AND WHEAT MILLING.

F

then passed through the "indent" machines, which remove corn-cockle and other small seeds and also the grains of other cereals, such as, oats, barley and rye. In addition, the wheat is brushed and scoured so as to remove lightly adhering dirt and thin skins, which are known as "beeswing."

As well as being subjected to these dry cleaning processes the grain is washed; the surplus water is removed in a centrifugal machine known as a "whizzer" and the damp wheat is then subjected to heat in a "conditioner." The object of this latter process is to bring the wheat to a physical condition which enables the endosperm to be separated from the skins more readily during the milling process.

The conversion of the cleaned and the conditioned wheat into flour on the modern flour mill consists broadly of a constant repetition of a series of five operations, namely, scraping endosperm out of the grain in granular form; removing the granular endosperm by sieving; purifying and grading the granular endosperm; grinding the endosperm to powder; removing the powdered endosperm by sieving. The initial scraping of endosperm from the split-open grains is performed on corrugated chilled-iron rolls which constitute the "break system." The number of breaks rolls in a modern mill is usually four or five. The removal of the granular endosperm from the broken open grains is done by "scalping," and is performed upon sieves or "plansifters" that operate in a horizontal plane, or on rotating cylindrical sieves, which are known as centrifugals. The separated granular endosperm, which is termed semolina, middlings or dunst according to its particle size, is graded and purified on machines known as "purifiers," which consist of a series of reciprocating sieves through which an upward current of air is passed. The grinding of the purified semolina and middlings to flour is performed upon smooth chilled-iron rolls, which constitute the "reduction system." The number of reduction rolls varies with the size of the mill but may reach sixteen. The powdered endosperm, i.e., the flour, is removed by sieving, the sieving machines being known as "flour dressers."

When the streams of flour leaving the various reduction rolls are all mixed together the resulting product is known as "straight-run" flour. Often, however, the purer stocks from a few of the reduction rolls at the head of the mill are separated from the

remaining reduction flours and are mixed together to form a
"patent" flour. Such flours represent a low extraction, e.g.,
30 per cent to 50 per cent, and sell at a higher price than straight-
run flour, which is of longer extraction.

Fig. 2 shows in diagramatic form the basic series of operations
involved in the flour milling process. This is, however, a very
simplified diagram, which omits a number of intermediate

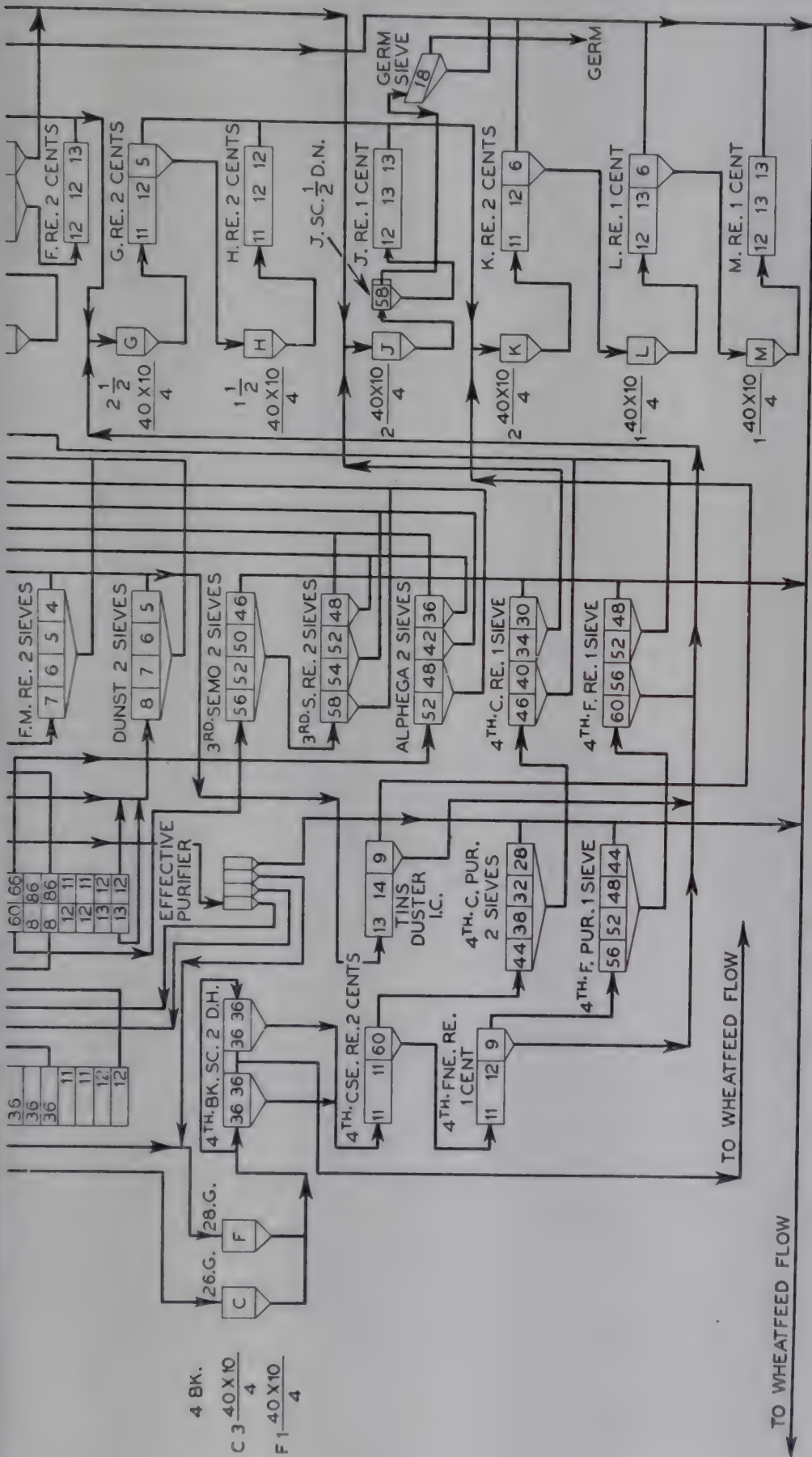FIG. 3.—PNEUMATIC INTAKE PLANT FOR TRANSFERRING WHEAT FROM SHIPS
TO THE MILL.

(Henry Simon, Ltd.)

A TLS. SIEVE 34 30

A. SC. 1½ D.H. 56 56 56 56

A. RE. 4 CENTS 10 11 11

B. RE. 3 CENTS 10 11 4

B2 RE. 1 CENT 11 12 60

C. RE. 5 CENTS 10 11 11

D. RE. 3 CENTS 11 12 5

E. RE. 2 CENTS 11 12 12

F. SC. 1 CENT 60 60 48

A

B

B2

C

D

E

F

ALL REDUCTION ROLLS 40X10

5 40X10 / 4

4 40X10 / 4

2 40X10 / 4

7 40X10 / 4

4 40X10 / 4

2 40X10 / 4

2 40X10 / 4

Y 1 40X10 / 4 32 GROOVES

TO 4TH. BK. SC.

A T.LS. PUR. 2 SIEVES 46 42 36 32

X PUR. 2 SIEVES 54 48 42 36

C.S. 3 SIEVES 32 28 24 18

M.S. 3 SIEVES 42 38 34 30

F.S. 4 SIEVES 54 50 44 38

C.M. 3 SIEVES 58 54 48 44

C.M. RE. 3 SIEVES 62 58 54 50

F.M. 2 SIEVES 6 5 4 3

X SC. 1 CENT 60 60 30

X

1ST. BK. GRADER ½ PLAN

1½ 40 X 10 / 4 30 GROOVE PLANS

FEED FROM GRADER

1ST. BK. SC. 1½ PLANS

2ND. BK. GRADER ½ PLAN

2ND. BK. SC. 1½ PLANS

3RD. BK.G. ½ PLAN

ALPHEGA

3RD. BK. SC. 2 PLAN

48 48 60 60 86 86 86 9 9 9 12 13

48 48 60 60 86 86 86 9 9 9 12 13

50 28½ PLAN 50 28 60 28 60 66

10W 10 20 20 36 36 36 36 10 10 11 11

12W 12 20 20 36 36 36 36 10 10 11 11

14 14 30 36

1 BK. C4 40 X 10 / 4 F2½ 40X10 / 4

8.G. 10.G. F C

2 BK. C4 40X10 / 4 F2½ 40 X 10 / 4

14.G. 16.G. F C

3 BK. C3½ 40 X 10 / 4 F1½ 40 X 10 / 4

20.G. 22.G. F C

FIG. 4.—FLOW SHEET OF 40-SACK FLOUR MILL.

(*"Flour Milling Technology"—Smith*)

operations and the re-treatment of various stocks. How complex the process becomes, because of the necessity to re-treat and sub-divide various stocks in order to obtain the maximum yield of flour of highest purity, is seen in Fig. 4, which is the flow sheet of a modern mill of moderate size.

Wheat Intake

According to the situation of the mill, the incoming wheat may arrive by ship, by barge, by railway truck or by road trans-port. The method of transferring the incoming grain into the large storage bins, or silos as they are called, will vary with the size of the mill and with the mode of transport. In small mills where wheat is arriving by road and rail the grain may be tipped into a pit or hopper which feeds a bucket elevator, that is, a series of bucket-like receptacles fitted to a moving band which pass through the wheat and carry it upwards. Wheat arriving at larger mills by water is conveyed from the ship to the silos by a pneumatic system in which the grain is sucked up a flexible pipe. Figs. 3 and 5 illustrate this pneumatic system of dealing with
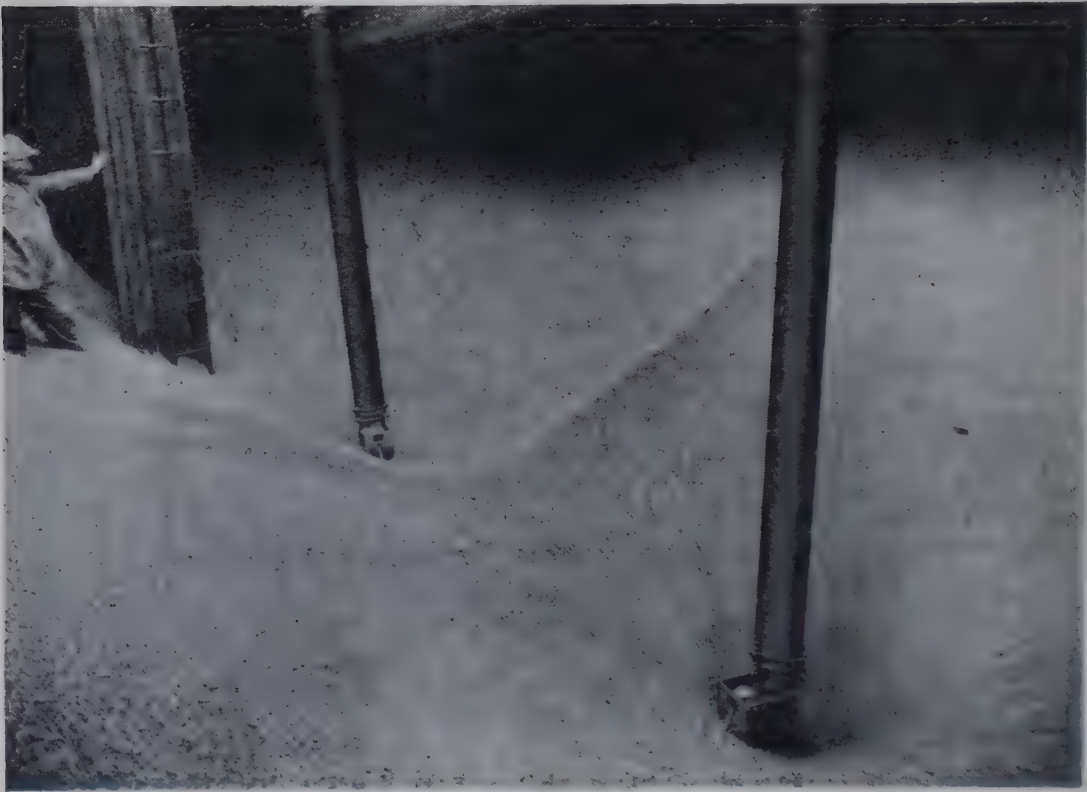
FIG. 5.—WHEAT BEING DISCHARGED FROM SHIP'S HOLD BY PNEUMATIC INTAKE.

(*Henry Simon, Ltd.*)

incoming wheat. When the wheat is discharged from the intake plant it passes through automatic weighing machines and thence to aspirated sieving machines which remove the coarser impurities and dust. After undergoing this preliminary cleaning the grain is transported on conveyors to the storage bins.

Wheat Cleaning

When wheat is required for milling it does not pass direct from the storage bins to the mill but is circuited through the "screen-room," where it is subjected to a vigorous cleaning and is then washed and conditioned.

The first operation in the screen-room is a sieving which is preceded or followed by an aspiration. The sieving machine is designed to remove miscellaneous impurities which are either larger or smaller than wheat grains. The grain can then with advantage be passed over a magnetic separator which will remove any scraps of metal that may be in the wheat.

The next stage of the wheat cleaning process is performed by disc separators. These consist of a series of metal discs with indented surfaces which rotate in a vertical plane inside a cylinder. The wheat is impelled along the cylinder by vanes attached to the central shaft and as the discs, which are partly buried in the mass of grain, rotate their indents pick up grain or seeds according to their dimensions and discharge them into troughs at a later stage of their travel. Some discs bear indents which will carry wheat but which will reject barley and oats, while the indents on other discs will remove seeds smaller than wheat but will reject the latter.

Washing and Conditioning

Despite the intensive dry cleaning operations which are applied in the screen-room, wheat may retain some types of impurities. These may include dirt which is adhering firmly to the wheat, and small stones and pellets of soil which are sufficiently similar to wheat in size to preclude their removal by sieving. These impurities can be effectively dealt with by washing. The washing is effected by feeding the wheat into a worm conveyor immersed in running water, the conveyor being so designed that the upward motion of the water which it produces enables the wheat to be carried the length of the worm without falling to the bottom of

the trough.   This treatment loosens and removes adhering dirt, while stones which may be present sink and are removed by another worm conveyor.   The washed wheat passes into a centrifugal machine known as a whizzer.   This consists of a number of inclined beaters on a vertical shaft which revolves at about 400 r.p.m. inside a stationary perforated casing.   On its passage upwards through the whizzer the wet wheat is thrown with considerable force against the perforated casing, whereby the unabsorbed surface moisture is removed.

From the whizzer the grain is conveyed to a conditioner, a machine in which the damp wheat is subjected to heat for the purpose of distributing the added moisture throughout the grain in such a manner that the separation of the endosperm from the skins and the sieving of the separated endosperm can be performed most efficiently.   The standard form of conditioner is a tall metal machine of rectangular section which is fitted with a large number of air ducts, hot-water radiators or both.   Wheat is fed into the top of the machine and descends slowly by gravity being heated in the earlier stages either by hot air or by the radiators. In the latter part of its travel the wheat is cooled by means of air currents.

In a modern development of the conditioning process wheat is wormed through a drum where it meets steam jets, whereby its temperature is raised to 120° F. to 160° F. in less than one minute. The wheat is then passed immediately into cold water.   It is claimed that the sudden rise and fall of temperature causes some loosening of the outer skins and thus aids the clean removal of the endosperm during the milling process.

Native wheat is often received at the mill with a moisture content too high for satisfactory milling and such wheat instead of being washed and conditioned in the normal manner has to be dried.   This can usually be done on the conditioner.

Before the conditioned wheat passes to the mill it is usually subjected to two final cleaning processes.   In the first of these the wheat is passed through a machine known as a scourer in which it is rubbed by beaters against the inside of a metal cylinder which often is lined with emery.   The final cleaning operation is brushing.   The wheat is conveyed along a cylinder by means of a spiral brush, the vigorous brushing which it receives loosening any remaining dust and dirt and giving the wheat a polish.

FIG. 6.—PLANSIFTERS IN A MODERN MILL.
(Henry Simon, Ltd.)
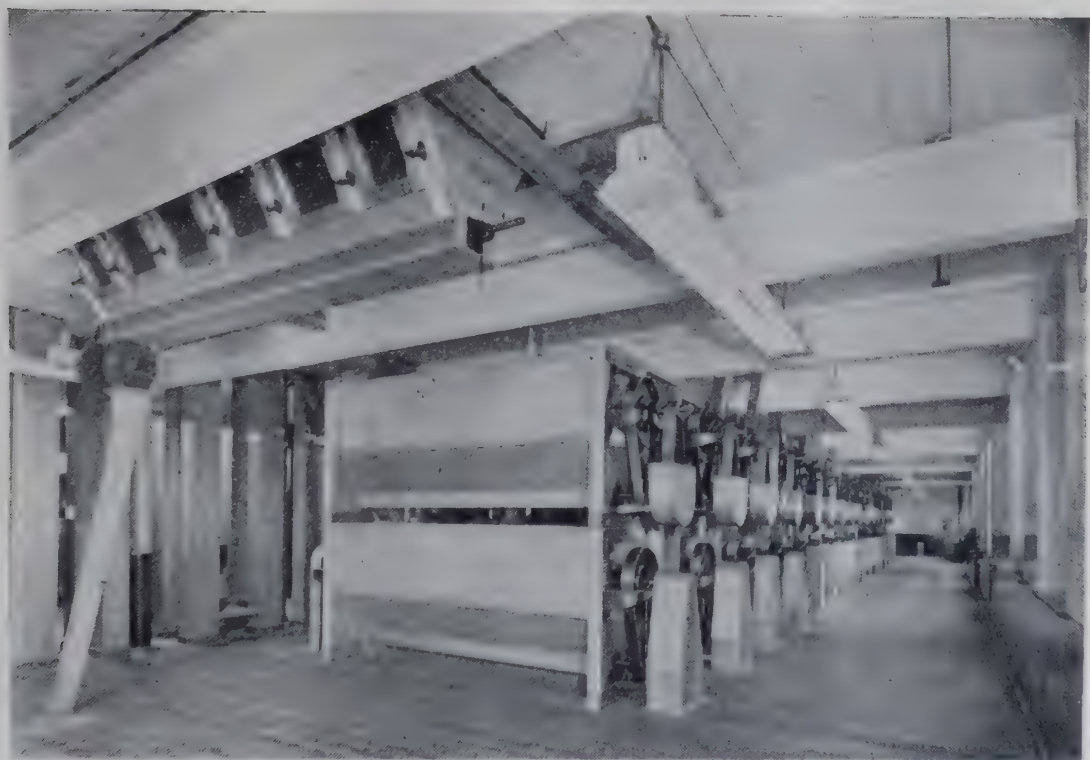
FIG. 7.—CENTRIFUGALS IN A MODERN MILL.
(Henry Simon, Ltd.)

The Break System

On entering the mill the conditioned wheat goes to a pair of corrugated chilled-iron rolls which rotate at a differential speed of 2½:1 thereby exerting a shearing action on the grain which passes between them. The effect of this action is to split open the grains and at the same time to scrape out some of the endosperm in the form of granules. At the same time a small amount of endosperm is released in powder form, that is as flour. The stock leaving these rolls, i.e., the mixture of split open grains, released granular endosperm and a very small amount of flour, is conveyed to the first break scalper where it is sieved into its component fractions.

The split open grains are then sent to the second break rolls, where they are opened up more or less completely and more granular endosperm and a little flour are released. This mixed stock is sieved and the split open grains go to the third break rolls where more endosperm is scraped from them. After another sieving the residue of the grains, which is now little more than flattened skins with some adhering endosperm, goes to the fourth break rolls, which in many mills perform the final scraping, although some mills operate a fifth break. The stock from the final break rolls goes to the corresponding scalper and the residue remaining after the released endosperm has been sieved away is bran.

Break Scalping

The separation of the stock leaving a break roll into its component fractions is accomplished by sieving on a centrifugal or on a plansifter. The former is a horizontal cylindrical framework covered with sieves which revolve around its axis, while the latter consists of a nest of sieves which rotate in a horizontal plane. Whichever method is used the outcome is the same, namely, the segregation of the broken open grains, the granular endosperm in the forms of semolina, middlings and dunst, and the small amount of break flour. The broken open grains from any break scalping are sent to the next set of break rolls, except in the case of the last break scalping where they go to the bran sack. The semolina and middlings are sent to purifiers, the dunst goes to the reduction system, while the small amount of break flour goes direct to the flour sack. Figs. 6 and 7 which are

photographs taken in mills show respectively the plansifters and
the centrifugals.

Purification

The semolina and middlings released by the break rolls have
to be "purified" before they pass to the rolls which will crush
them into flour.   A purifier by means of which this is accom-
plished consists of an inclined reciprocating sieve, the mesh size of
which increases from the head to the tail of the machine.   The
sieve is enclosed in a casing and air is blown up through it.   As
the granular endosperm is conveyed down the sieve by the
reciprocating action, it is graded because of the different mesh
sizes over which it passes and, at the same time, small pieces of
wheat skin, which would otherwise pass through the sieve and
thus contaminate the sieved stock, are removed by the upward
current of air.   Larger pieces of skin are held up by the air current
and float along to the end of the machine where they are removed.
Fig. 8 shows the purifier floor of a mill.

Reduction System

The function of this part of the mill is to pulverise the graded
and purified semolina and middlings to the fineness of flour and
at the same time to flatten any pieces of skin which may be
present.   The reduction is performed upon pairs of smooth

FIG. 8.—PURIFIERS IN A MODERN MILL.
(Henry Simon, Ltd.)

chilled-iron rolls which revolve at a differential speed of $1\frac{1}{4}$:1. The stock leaving the rolls is sieved in order to remove the flour which has been made and also flattened pieces of skin, and the endosperm which is still not fine enough to be flour is sent to another pair of reduction rolls. Stock from which endosperm could not be removed in a reasonable state of purity even after further treatment on reduction rolls is sent to the wheat feed. This process of grinding on reduction rolls and sieving is repeated until the required amount of flour has been obtained. The sieving of the flour from the as yet insufficiently ground semolina or middlings is performed upon centrifugals or plansifters. The roller floor of a mill, which houses both the break and the reduction rolls, is shown in Fig. 9.

Pneumatic Conveying

The conversion of wheat into flour can be summarised broadly as numerous repetitions of two operations, grinding and sieving and it follows, therefore, that during the milling process the many intermediate stocks have to be conveyed from one machine to another, and indeed from one part of the building to another. It has for long been the standard practice to utilise gravity or bucket elevators for the vertical transport of stocks and worms or moving belts or chains for horizontal conveyance. In recent years, however, systems of conveying stocks within the mill by means of air currents have been developed and undoubtedly pneumatic conveying will become widely used in the future. In Fig. 10 is seen the floor of a mill equipped with pneumatic conveying.

Advantages claimed for pneumatic conveying are:

1. Cooler rolls and stocks which makes for more efficient sieving.
2. Reduction in the dust content of the atmosphere and avoidance of sweating.
3. Reduction in infestation.
4. Reduced fire risk through replacement of wooden spouting by metal.

Flour Treatment and Flour Bleaching

When freshly milled flour is stored it undergoes an ageing effect which whitens its colour and at the same time causes it

FIG. 9.—THE ROLLER FLOOR OF A MODERN MILL.
(Henry Simon, Ltd.)
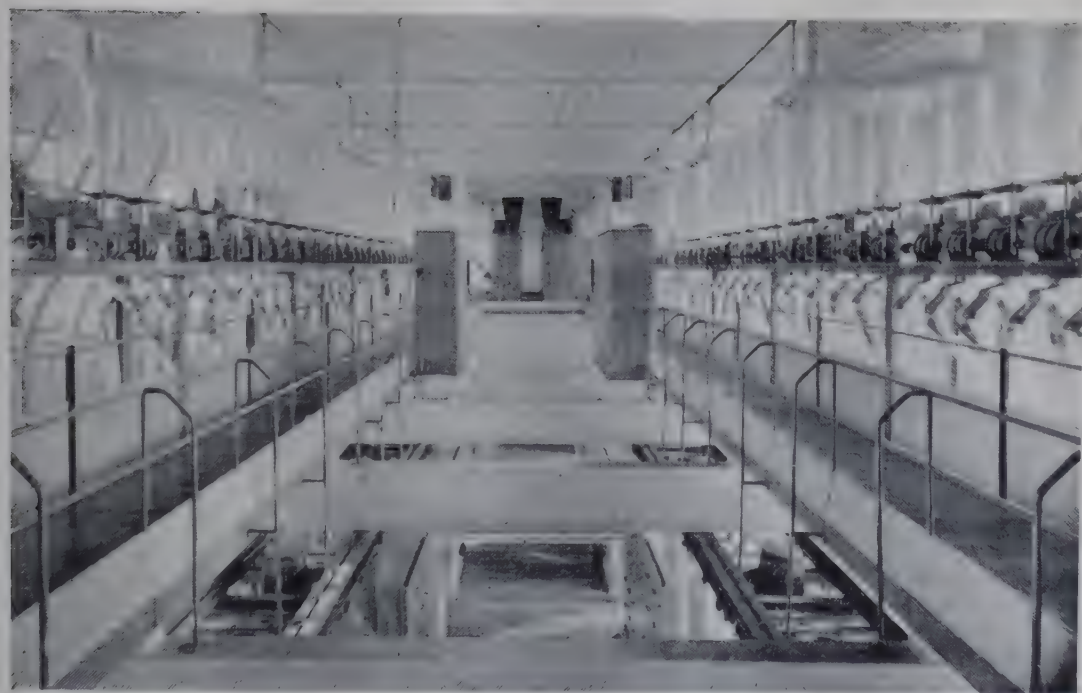
FIG. 10.—THE TOP FLOOR OF A MODERN MILL EQUIPPED WITH
PNEUMATIC CONVEYING.

(Henry Simon, Ltd.)

to produce a stronger and more resilient dough when mixed with water, and hence a dough that handles better in the bakery and gives a bolder loaf.   Because of this it was at one time customary for the miller to give his flour several weeks' ageing before he sent it to the baker.   Subsequently it was discovered that the improvement in dough properties and the bleaching, which were obtained slowly by natural ageing, could be produced rapidly by adding to the flour certain oxidising substances.   Some of these compounds, such as, ammonium persulphate and potassium bromate, improve the baking quality but have no effect upon the whiteness; others, such as nitrogen peroxide and benzoyl peroxide, bleach the flour but have no effect upon dough properties; while yet others, as, for example, nitrogen trichloride and chlorine dioxide, both bleach and "improve."

It is now the standard practice for millers to use one or a combination of these substances to produce the necessary ageing effect and to bleach their flour at the time of manufacture and thus avoid the disadvantages of protracted storage.   The solid improvers and bleachers suitably diluted are fed in powder form into the flour by means of specially designed feeders, while the gaseous forms of treatment are applied by metering the gas into an agitator in which the flour is thrown up as a cloud as it passes through.

The proportions in which the improvers and bleachers are normally used are:

Nitrogen peroxide	5 parts per million
Benzoyl peroxide	15–45 parts per million
Ammonium persulphate	160 parts per million
Potassium bromate	20 parts per million
Nitrogen trichloride	60 parts per million
Chlorine dioxide	30 parts per million

COMPOSITION OF WHEAT AND ITS MILLED PRODUCTS

The chemical composition of wheat varies according to the variety and to the conditions under which it is grown and harvested.   Thus, different strains of wheat grown side by side may vary markedly in composition, and similarly wide differences in composition may be encountered in samples of the same strain of

wheat grown under different conditions of soil or climate. The range of variation that may be expected is shown in Table I.

The chemical composition of a flour will depend upon the wheat from which it was milled and also upon the extraction; the higher the extraction of a flour produced from a given wheat the higher its contents of protein, mineral matter and fibre. The approximate compositions of different types of flour are shown in Table II.

The bran and the sharps, the two main by-products of flour milling, have the approximate compositions shown in Table III.

The other by-product of the flour mill, germ, has the approximate composition shown in Table IV.

During the war the nutritional status of flour and bread assumed considerable importance and was intensively studied. Particular attention was paid to the vitamin and iron contents at different levels of extraction. Representative figures for different types of flour are given in Table V.

TABLE I

APPROXIMATE RANGE OF VARIATION IN COMPOSITION OF WHEAT

Moisture . . .	8%–20%
Protein . . .	8%–15%
Fat	1·5%–2·5%
Mineral matter . .	1·5%–2%
Fibre . . .	2%–2·5%
Carbohydrates . .	65%–78%

TABLE II

APPROXIMATE COMPOSITIONS OF DIFFERENT TYPES OF FLOUR

	Flour of 72% Extraction per cent	Flour of 80% Extraction per cent	Flour of 85% Extraction per cent	Wholemeal (approx. 95% Extraction) per cent
Moisture . .	13–15·5	13–14·5	13–14	13–13·5
Protein . .	8–12·5	9–12·5	9–13·5	10–14
Fat . . .	0·8–1·5	1·1–1·7	1·5–2·0	1·7–2·3
Mineral matter .	0·4–0·5	0·55–0·65	0·7–0·9	1·4–1·6
Fibre . .	Trace–0·2	0·15–0·25	0·4–0·8	1·6–2·1
Carbohydrates .	70–78	70–76	69–75	66–72

TABLE III

APPROXIMATE COMPOSITIONS OF MILLING BY-PRODUCTS

	Bran per cent	Sharps per cent
Moisture . .	13·5–14·5	13·5–14·5
Protein . .	12·5–15·5	13–16
Fat . . .	3·5–4·5	4·5–5·5
Mineral matter .	5·0–6·0	3·0–4·5
Fibre . .	9·0–12·0	4·0–6·0
Carbohydrates .	47–56	55–62

TABLE IV

APPROXIMATE COMPOSITION OF COMMERCIAL WHEAT GERM

	per cent
Moisture . . .	10–13
Protein . . .	22–30
Fat	6–11
Mineral matter . .	4–5
Fibre. . .	1·8–2·5
Carbohydrates . .	38–56

TABLE V

APPROXIMATE NUTRIENT CONTENTS OF FLOURS OF DIFFERENT EXTRACTIONS

Extraction	50% (Patent Flour) mg/100g	72% mg/100g	80% mg/100g	85% mg/100g	95–100% (Whole meal) mg/100g
Thiamine . .	0·08	0·10	0·25	0·30	0·40
Riboflavin . .	0·05	0·06	0·07	0·10	0·13
Nicotinic acid .	0·70	0·80	1·60	1·90	5·50
Pantothenic acid .	0·40	0·60	0·90	1·10	1·50
Pyridoxin . .	0·10	0·15	0·25	0·30	0·50
Iron . . .	1·00	1·50	1·70	2·20	3·00

The riboflavin, pantothenic acid and pyridoxin are found in greatest concentration in the aleurone layer and the endosperm, the nicotinic acid in the aleurone layer, and the thiamine in the scutellum and aleurone layer. The germ has a relatively high vitamin E content but vitamin A is not present in significant amount in wheat.

When the milling industry was decontrolled in September 1953, a nutritional standard for flour was adopted, this standard requiring that each 100 g. of flour should contain 0·24 mg. thiamine, 1·60 mg. nicotinic acid and 1·65 mg. iron. The standard can be attained by milling a straight run flour at 80 per cent extraction but millers are permitted to produce flours of lower extraction, including patent flours, provided they make good the nutritional deficiences by adding the requisite amounts of thiamine, nicotinic acid and iron. They can do this by purchasing a standard "master mix" and adding it to their white flour in prescribed proportions. The composition of the master mix is:

Thiamine	0·21 g/oz.
Nicotinic acid . . .	1·00 g/oz.
Iron (*ferrum redactum*) . .	0·89 g/oz.
Dry flour	26·25 g/oz.

and it has to be added to 70 to 72 per cent extraction flour in a proportion of 1 oz. per 280 lb. and to each 280 lb. of patent flour in a proportion of 1·1/10th oz.

The practice was adopted during the war of adding calcium in the form of *creta praeparata* to flour as a means of increasing the calcium content of the national diet. Millers are still required to make this addition at a level of 14 oz. of *creta* per 280 lb. of flour.

BAKING QUALITY OF WHEAT FLOUR

Protein Quality

When wheat flour is mixed with about half its weight of water a dough is formed which has elastic properties. In this respect wheat flour differs from all other cereal flours, because, on admixture with water, they yield plastic masses which are not

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elastic. The reason for this difference is that in the presence of water the two main proteins of wheat flour form a complex, known as gluten, which is both extensible and elastic. During dough formation the proteins dispersed throughout the flour are converted into a network of interwoven strands of gluten and this serves as the skeleton girder work of the dough mass. Obviously the physical properties of the dough will depend upon the number of gluten girders which are present and upon the properties possessed by these girders. These two factors depend in turn upon the proportion of protein in the flour and upon the nature of that protein. As wheats may differ widely not only in protein content but also in protein quality, the dough properties of a flour are determined by the wheats from which it is milled. Some wheats yield a gluten which is strong and tough, while others give rise to a weak and flowy gluten.

The physical properties required in a dough depend upon the purpose for which the dough is to be used and this, therefore, determines the quantity and quality of protein called for in the flour. If bread is to be made, the flour should contain a reasonably high proportion of protein which furnishes a strong and elastic gluten. Flour intended for confectionery work should be only moderately proteinous and should yield a weaker and less elastic gluten, while biscuit flours should be low in protein content and the glutens they furnish should be weak and extensible.

Diastatic Activity

The basic principle of the breadmaking process is to make a dough from wheat flour, to inflate that dough with gas, and then to cook and "set" the aerated mass. If bread of good quality is to be obtained, it is necessary firstly that an adequate supply of gas shall be produced within the dough, and secondly, that the dough shall expand to a satisfactory extent under the pressure of that gas. The extent to which this latter requirement is fulfilled is related to the physical properties of the dough and these, as explained in the preceding paragraph, are determined by the quantity of protein in the flour and its physical properties.

The other factor in the production of good bread—gas production—is also related to the nature of the flour. The gas is produced within the dough by the action of added yeast upon

sugar, and good bread will result only if sufficient sugar is available to the yeast to ensure an adequate supply of gas throughout the fermentation period. The sugars which are naturally present in flour are not sufficient for this but fortunately flour contains diastatic enzymes, compounds which during the fermentation convert some of the starch of flour into sugars. The diastatic power of a bread flour must, therefore, reach such a level that it furnishes a sufficient reserve supply of sugar during the fermentation of the dough.

While the diastatic activity of a flour must be sufficient to supply the needs of the yeast for sugar, too high a diastatic activity is detrimental to bread quality. Associated with the diastatic enzyme which converts starch into sugar is one which turns starch into dextrins, which are sticky substances. If dextrin production is too great the crumb of the loaf will be sticky.

WHEAT BLENDING

The production of good flour depends upon two things—the skilful application of sound milling principles and the choice of a suitable wheat blend. No matter how efficiently the milling process is performed, a good flour will not be obtained if the wheat blend is at fault.

The quantity of wheat we grow in Britain is sufficient to meet only about 30 per cent of our requirements and hence a high proportion of imported wheats has to be used in the mill. These wheats differ widely in protein content, protein quality and diastatic activity, and it behoves the miller so to blend them in relation to these three factors that the resulting flour will give a dough of suitable physical properties which possesses satisfactory diastatic power. If the miller is to do this successfully he must be supplied in advance with the relevant chemical and physical data appertaining to the wheats he proposes to use. How great is the need for this advance information is revealed in Table VI which gives the minimum and maximum figures for a range of samples of several types of wheat examined during one month recently in the author's laboratory. The stability and strength figures, which are a measure of the gluten quality, were obtained by means of an Alveograph, an instrument which will be

described later.   It will be noted that the data of different types of wheat differ greatly but also that there may be a considerable range of variation in the figures for any one type.

TABLE VI

MINIMUM, MAXIMUM AND AVERAGE FIGURES FOR SAMPLES OF DIFFERENT TYPES OF WHEAT AVAILABLE TO MILLERS DURING A PERIOD OF ONE MONTH

	Protein Content	Stability of Gluten	Strength of Gluten	Diastatic Activity
	per cent			
No. 1 Manitoban				
Average	12·37	111	75	1·75
Minimum	11·57	92	60	1·60
Maximum	12·77	125	90	2·10
No. 3 Manitoban				
Average	12·20	96	61	1·70
Minimum	11·86	89	48	1·55
Maximum	12·77	115	85	1·80
Hard Winter				
Average	10·72	67	43	1·65
Minimum	9·98	53	33	1·55
Maximum	11·97	99	68	1·85
Argentine				
Average	13·40	97	63	1·60
Minimum	11·24	74	39	1·40
Maximum	14·54	125	98	1·85
Australian				
Average	9·35	81	30	1·65
Minimum	9·29	70	26	1·55
Maximum	9·41	90	36	1·80
English				
Average	9·35	52	20	1·65
Minimum	7·58	35	10	1·35
Maximum	11·17	110	52	1·90

The broad principle on which a miller usually works when formulating his blend for the production of bread flour is to utilise a suitable mixture of strong and weak wheats as the basis of his blend and then to dilute that mixture with wheats of intermediate strength. These latter wheats, which are known as "filler" wheats, are not sufficiently strong to carry any weaker wheat but, on the other hand, are strong enough to need no support. The chief wheat used in the strong group of the blend is Manitoban but Garnet and Northern Spring may also find a place there. The main ingredient of the weak wheat group is English, although Australian, Continental, and some weak Canadian and American wheats may be included. Typical "fillers" are some types of Argentine wheat, more particularly Rosafe, and Hard Winter.

When the quality of the protein is under consideration, it is not only the strengths of the proteins of the wheats available which have to be taken into account. Attention must be paid to other physical properties of the proteins and to the balance between them. If a protein of one wheat shows an excess or deficiency of a given property, then the blend of wheats should be so arranged that the defect is remedied by the inclusion of a suitable proportion of a wheat of a compensatory nature.

If a miller intends to produce flour for confectionery work or flour for domestic use, often termed "scaling" flour, he would use a blend which would yield a less proteinous flour than a bread flour and would ensure that the protein was weaker in nature. To do this he would employ a smaller proportion of strong wheats and a higher proportion of weak wheat. Whether he employed fillers would depend upon the wheat situation.

When flour for biscuit factories is to be made, it is customary for the miller to use nothing but English wheat. He must, however, select this with care because many types of even English wheat are too strong and too proteinous to make good biscuit flour.

WHEAT TESTING

Although the British miller, who is accustomed to using wheats from all parts of the world, is conversant with the general standard of quality of any one type of wheat, he can only ensure

that a proposed wheat blend will prove to be satisfactory by knowing in advance the relevant chemical and physical data relating to the particular cargoes he has purchased.   As Table VI shows, at a time when the mean strength figure of No. 3 Manitoban is 61, a particular cargo may show a strength of only 48. A miller who had purchased the latter wheat and was unaware that its strength was significantly below the average might formulate a blend containing it which was satisfactory on paper but would furnish a flour that would be criticised by the baker on the score of weakness.   It is, therefore, customary for British millers to arrange that they are provided with reports upon all imported and some native wheats before those wheats are used.   A few large mills obtain the information from their own laboratories but most millers employ the services of a consultant who, with his large and extensively equipped laboratories, is able to deal promptly with large numbers of wheat samples.

The object of a scheme of wheat testing should be to provide the miller with a numerical assessment of each of those properties of a wheat which he needs to take into consideration when formulating his blend and, when there is an excess or lack of any property, to advise him what type of wheat would best serve as a corrective in the blend.   This object can be achieved if the wheat is tested for moisture content, protein content and diastatic activity, and the physical properties of its protein are reliably assessed on a numerical basis.   Methods of making these tests are discussed on p. 211–220.

<div align="right">A. J. A.</div>

BREADMAKING

BREAD has been the oldest and most general food of the world ever since man acquired the herd instinct, settled in groups more or less permanently in one spot and started to cultivate the ground. The fundamental materials are easy to obtain: wheat flour, salt, yeast and water and the making of bread—of a sort—is a relatively simple process.

INGREDIENTS OF BREAD

It cannot be said that there is any one flour which, used by itself alone, is ideal for this purpose and the flours from different countries show big differences in their value as breadmaking flours. Moreover, tastes in different countries and even in the same country are for different flavours and types of bread. Breadmaking has grown, therefore, from a simple household piece of cooking to a complicated science.

To remedy these differences in the natures of the flours used and to satisfy regional tastes, bakers accordingly use one or more other materials besides the four basic ones. These may be such things as fat, malt extract and other enrichers, fermentation controllers usually denominated by the term "yeast foods" and, in the last ten years or so, vitamins and other accessory factors.

Proportions of Materials Used

The first step in the process consists in making as uniform a mixture as possible of the various ingredients. The actual proportions of the materials used depend on the kind of bread it is proposed to make, e.g., oven bottom or tin bread; the locality in which the bakery is placed (Scotland requires a different type of bread from Lancashire, Lancashire bread would not be popular in Birmingham, and none of the three would sell well in, say, London or South Wales) and the process to which it is to be subjected. This may be a short process of 2 to 3 hours, a longer one of 4 to

6 hours or the method known as the sponge and dough, in which part of the ingredients are allowed to ferment for a certain time before the dough is completed by the addition of the remainder of the ingredients.

The variations in process adopted are very numerous; even in the same town, supplying the same type of palate, bread will be made in several different ways, according to the ideas of the individual master baker. The variations in the quantities used and in the kinds of bread marketed are not, however, very great as a rule.

PREPARATION OF THE DOUGH

The first part of the process is the most important. A dough is a very stiff and difficult material to do anything with when it is made and any error in the quantities of the ingredients used cannot be easily rectified. Moreover, the salt, yeast, and other ingredients added to the water and flour are only about 1 to 2 per cent of the total of the latter ingredients and it is essential for success that the dough be uniform in composition. The flour is put into the bowl of the kneading machine and its temperature taken. The dough-maker requires his finished dough to be at a certain temperature and from the temperature of the flour he can either calculate or read off on prepared tables the temperature at which he should take the water. This latter is contained in a "tempering" tank which is fitted with a thermometer and marked off in gallons and fractions thereof, and fitted with a hot and cold water supply.

A gallon or two is drawn off first into one or two containers. In one the yeast is thinned down to a liquid, in another the salt or mineral improver is dissolved or worked into a suspension and any other ingredient used in small quantity is treated in the same way. Malt extract should be thinned down, milk powder worked into a cream, and so on. The kneader is started and the remaining water is then added steadily to the flour in the bowl. The small quantities of yeast, and other ingredients are poured into the bowl and when all are added the dough is kneaded for about 10 to 20 minutes. The finished dough should be smooth and "clear." To decide when this has been done is where the art of the dough-maker comes in.

Fermentation of the Dough

After making, the dough is either allowed to remain in the pan, if this is removable, or transferred to a trough, covered with a cloth to prevent draughts which may cause a skin to form and placed in a part of the bakery where the temperature is reasonably uniform. There it is allowed to ferment for a pre-determined period and is only disturbed for the process of "knocking back" referred to below.

What is there in a Dough?

Though this is quite well understood in a general way, it is impossible to give a complete answer to this question. It must be borne in mind that there are an enormous number of different substances in a dough in its first stage and most of them can have some effect on the final result. Flour contains water, proteins, fat, carbohydrates, salts and other substances in small quantities which are not without some action on fermentation. Salt is a reasonably pure substance but water normally contains dissolved salts, some of which are essential for a good fermentation. Yeast today is almost a 100 per cent standardised product and that helps to simplify control. Finally, there are bacteria in flour and one cannot prevent bacteria from the air entering the dough. Some of these are of importance during the fermentation period.

What Happens in the Dough during Fermentation?

Here again it is impossible to give a complete answer to this question but the main changes are two in number:

(a) THE AERATION OF THE DOUGH. Yeast is capable of decomposing sugars with the production of carbon dioxide and alcohol. The latter is of no value in the bread and is almost entirely, but not completely, expelled from the dough in the oven. This change is not, however, a simple one and does not take place in one stage. Secondary reactions take place and numerous bodies have been identified in small quantities in dough. The carbon dioxide produced renders the dough acid and aerates it; the small bubbles permeate its mass, making the network which is characteristic of a cut loaf.

Without aeration, bread would be an extremely heavy and unpleasant food to eat and difficult to digest. More carbon dioxide is produced, however, than is necessary merely to aerate

the dough and inasmuch as all the carbon dioxide and alcohol produced come from the flour and therefore represent so much flour solids, and are subsequently expelled into the air, thus being entirely lost as food, it is clear that the reasons for a longer fermentation than is necessary for aeration alone must be good ones.

(b) THE MATURING OF THE DOUGH. When water is added to flour and a dough made, the proteins combine to produce a complex called "gluten." Wheat flour is unique in this respect and this is the reason why it makes better bread than flour from any other cereal. When first made, however, gluten is not in the ideal condition to form a skeleton or framework which can be stretched without breaking and will support the network of gas bubbles produced by the action of yeast, thus producing a bold and light loaf. This "maturing" action is even more important than aeration, which can be achieved by other methods, e.g., baking powders, or forcing a gas into the dough under pressure as was actually done on a commercial scale. A comparison of the flavour and character of yeast-raised and baking powder-raised loaves shows the superiority of yeast-raised bread.

The Factors Affecting Gluten Maturing

This is important enough to be considered under a separate heading. While all the factors affecting this maturing are not known, the important ones are recognised and the chief one is the hydrogen ion concentration of the dough. As mentioned above, the dough is rendered acid by the carbon dioxide present and this affects the maturing very favourably.

Another influence at work in long doughs is the proteolytic enzymes, which have the power of breaking down complicated proteins such as glutenin and gliadin (of which gluten is mainly composed) to less complex proteins. This process of degradation must not proceed too far, otherwise the gluten will lose its character nor, equally, must the acids be allowed to act excessively on the dough, otherwise the dough will be "over"-fermented and will make bread of a squat, heavy and unappetising appearance.

It has also been found that certain mineral salts have an accelerating action as regards gluten maturing, so that instead of allowing 2 to 3 hours for the formation of these acids and for their action and that of the enzymes in the gluten to take place, the time

can be reduced by 20 per cent and more. Inasmuch as, during all this time, yeast is at the same time destroying flour solids and producing carbon dioxide and alcohol from them, it can be seen that the use of these maturing agents results in an appreciable saving of flour, with a resultant greater yield of bread from the sack of flour.

"Knocking" or "Cutting-back" the Dough

Should the fermentation period be over two hours, it is a common and beneficial practice to "cut-back" the dough. This process consists in turning over and squeezing the dough so as to reduce its volume. The accumulation of alcohol in the interior of a big mass of dough tends to kill the yeast and by turning it over in this way, a certain amount of the alcohol is driven off, fresh surfaces are exposed to the air, a certain amount of air occluded in the dough, and parts of the dough which may not have been in contact with the yeast have a chance of being acted upon. The actual time or times when this cutting-back is carried out vary according to the length of the process and the preferences of the master baker, but in a four-hour process would normally be between $2\frac{1}{4}$ and $3\frac{1}{4}$ hours.

After-Fermentation Treatment

The dough being properly fermented, i.e., when the gluten has matured—this being ascertained by the feel and an examination of the way it stretches—it then passes on to the scaling or dividing process. From the fermentation stage onwards in large bakeries the remaining processes are automatic, and very little variation is possible. The importance of preparing the dough correctly and of fermenting it to the correct stage cannot, therefore, be stressed too strongly.

Dividing the Dough into Loaves

In small bakeries the dough is "scaled," i.e., weighed by hand; in larger bakeries the dividing of the dough is done by machinery, depending on delivery of a certain volume of dough. Dividers are adjustable and can be made to deliver any required volume of dough. Until the last war loaves had by statute to be one pound in weight or multiples of one pound. This is not the law now but weights are standardised and to ensure this the weight of

the dough to be taken is about one-eighth more than the finished loaf, e.g., to make a one-pound loaf take one pound two ounces of dough. This additional weight allows for the loss of weight in the oven due to evaporation of water and the expulsion of alcohol and some of the gas.

The " Hander-up "

After dividing, the dough travels to the next machine, the "hander-up." The "hander-up" is a machine for moulding roughly the dough pieces. At this stage the dough is in a very delicate condition and requires a little rest to recover from the harsh treatment of the divider, which has squeezed and cut it and handled it generally rather roughly. The cut edges want "healing," and it is the purpose of the "hander-up" to close up these cuts and to form a skin all round the piece. In this form, the piece is in the best condition to recover. This period of rest is called "intermediate proof" and lasts for 10 to 20 minutes.

The " Moulder "

After recovery, the pieces go through a "moulder," in which it again undergoes a gentle squeezing and pressing action. This renders the condition uniform throughout the piece, so that during the last half hour or so of fermentation (called the "final proof") the expansion and gas formation will be regular. In some bakeries, the same machine serves as both hander-up and moulder. The only essential difference in their action is that the moulder handles the dough much more gently than the hander-up. Both of them tend to set up a state of tension in the gluten net-work, rendering oven expansion and texture even throughout the loaf.

THE BAKING PROCESS

The Action of the Oven

The dough has now arrived at the oven and the actual trans-formation into bread is to take place. The normal temperature for a bread oven is 450° F. to 500° F. and the time of baking is from 40 to 50 minutes for a 2 lb. loaf. With brown and other breads the temperature is usually lower and the time longer.

STRAIGHT DOUGH METHOD   SPONGE and DOUGH METHOD

PREPARATION of INGREDIENTS
Weighing
Sifting and Blending Flour
Tempering of Water
Preliminary Mixing of Yeast
Salt, etc

PREPARATION of INGREDIENTS
for SPONGE
Weighing
Sifting and Blending Flour
Tempering of Water
Preliminary Mixing of Yeast
(Salt on occasion)

DOUGH MIXED

SPONGE MIXED

DOUGH
ALLOWED TO
FERMENT

SPONGE ALLOWED TO MATURE

SPONGE BROKEN UP and
MIXED WITH DOUGH INGREDIENTS

DOUGH ALLOWED TO FERMENT

DOUGH KNOCKED BACK

DOUGH KNOCKED BACK ON OCCASION

DIVIDING and SCALING

HANDING UP

INTERMEDIATE PROOF

FINAL MOULDING

TINNING UP

FINAL PROOF

BAKING

COOLING

SLICING and WRAPPING

COUNTER SALES, ROUNDS, WHOLESALE

FIG. 1.—PRINCIPAL METHODS OF BREADMAKING

Summary of the total production process in which a comparison is made
between the straight dough and sponge methods.

(*The Biscuit Maker & Plant Baker*)
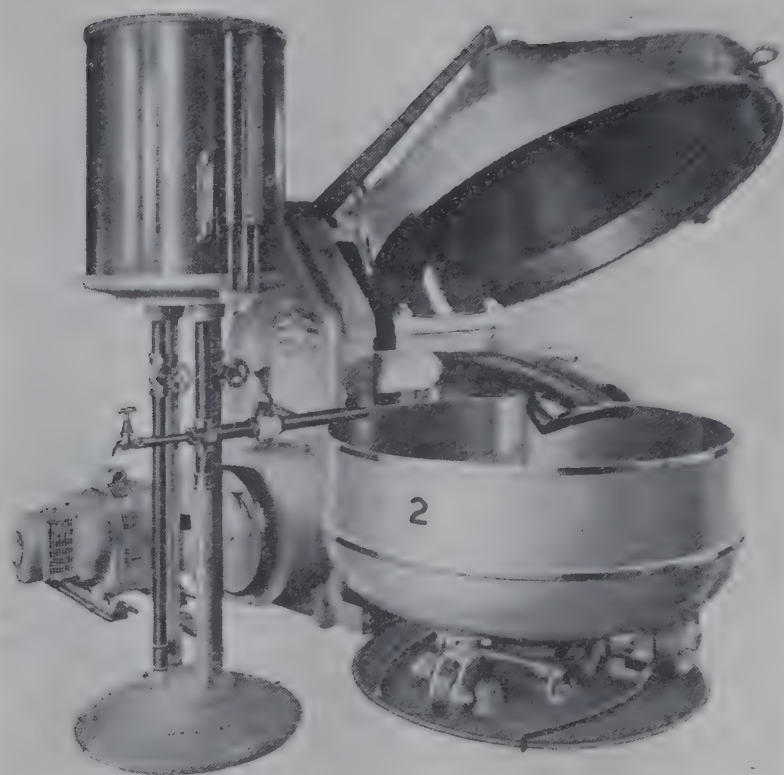
FIG. 3.—LOAF-MAKING PLANT, CONSISTING OF DIVIDER, FIRST PROVER,
AND FINAL MOULDER.
(Thomas Collins & Co., Ltd.)

Yeast Activity Increases as the Temperature Rises

The main changes that take place in the oven are as follows. For about the first 15 minutes the temperature does not rise above the point at which yeast ceases to be active. If the preceding stages have been carried out accurately, the dough arrives full of life at the oven and as the temperature increases this yeast activity is also very considerably increased.

The loaf can be seen to rise, owing to the greater development of carbon dioxide and expansion of the gas already contained in it, until it reaches the well-known dimensions of a 2 lb. loaf.

The temperature of the interior of a loaf never exceeds 212° F., but the exterior can, of course, go much higher. The first stage is the drying out of the outer surface of the dough and the formation of an impermeable skin.

Why Steam is Injected into the Oven

Up to this point steam can pass out of the interior of the loaf and the loaf can expand. The presence of this steam keeps the skin of the dough flexible. Until steam is produced from the loaf itself, it is customary to inject steam into the oven. All this while the gluten is coagulating and becoming "set," thus forming a framework for the whole of the loaf.

The Death Point of the Yeast

About the time the skin is formed, the death point of the yeast is nearly reached and expansion stops. If the fermentation has been correctly carried out, the loaf will keep the shape obtained at this point, because the gluten will be strong and able to support it.

Faults Due to Incorrect Fermentation

If, unfortunately, the loaf has suffered from incorrect fermentation, faults of several kinds will appear, tears in the side of the top crust, later on "flying tops," which is a sufficiently descriptive term not to require further definition. In the interior of the loaf the heat is gelatinising the starch and making it more easy to digest. It should be mentioned here that there is never sufficient water in a loaf to enable the whole of the starch to be gelatinised, and under the microscope it is possible to see in bread individual starch grains.

Why Loaves are a Pleasant Brown Colour

When the crust is dried out, it begins to caramelise. The pleasant brown colour of a loaf is due mainly to caramelisation of the sugar contained in the dough. Now it must be remembered that sugar is one of the important foods that yeast feeds on. There is a certain amount of sugar naturally in the flour, but the bulk of it is obtained by the action on starch of an enzyme called diastase, present in smaller or greater degree in the flour. The yeast may use up the sugar, however, more quickly than the diastase can produce it, and in some cases there will be a shortage of sugar in the dough when it goes to the oven.

This will be manifested by a paleness in the crust, the loaf is said to have a poor "bloom," and this detracts from its appearance, a serious point when it comes to selling the loaf.

At the end of three-quarters of an hour, the starch has reached its maximum gelatinisation, the gluten is all coagulated, the outer crust formed firmly all round—the loaf is made.

BREAD WRAPPING

In bakeries, where bread is wrapped, care is still necessary. On cooling, the solid crust contracts and small cracks appear through which steam can escape from the interior. If bread is wrapped warm, it will be maintained for several hours in a warm moist atmosphere, which is the ideal condition for the growth of moulds and the development of that bogey of the bread-trade, "rope." It also results in the formation of a soggy crust. It is, therefore, necessary to allow the bread to cool to an extent which will prevent the formation of these conditions.

F. E. T.
W. P. F.

CHAPTER 9

BISCUIT MANUFACTURE AND CAKE MAKING

1. BISCUITS

Biscuit doughs are made mainly from flour, fat, sugar and water, with minor amounts of flavours, colours and aerating agents and, possibly eggs and fruit. The doughs are formed to the required shape by one of various processes and baked in travelling ovens to a moisture content of 1 to 4 per cent.

INGREDIENTS

Flour

The principal flours are milled from English soft wheats, Canadian Spring wheats, Canadian Winter wheats and Australian wheats. American soft wheats and Argentinian wheats may also be used if the currency position permits them. The baking character of flour is the largest variable among the raw ingredients and it is with the English flours that the main variations occur.

Sword[1] lists some seventy wheats grown in Britain which he has examined; seven of which are consistently suitable for biscuits and seventeen frequently suitable. Variations in quality arise from (a) differing climatic conditions from locality to locality and from year to year, (b) possible preliminary drying by the farmer, (c) different conditioning of the wheat before it is milled and (d) different systems of milling.[2]

The requirements for a suitable English flour are difficult to stipulate objectively but it is generally agreed that the protein content should be low (under 8.5 per cent) and that doughs made from the flour should be easily extensible, but not elastic, and should flow somewhat in the oven. A determination of the protein or of the complex known as gluten (a mixture of protein, fat, fibre and starch obtained by washing out as much starch as possible from the dough) is next to useless as a test for suitability. This is because not only the quantity of protein is important but also its character, and this latter is not determinable chemically

H

The only really satisfactory method is to bake several stone of dough in the bakehouse but, as this may prove expensive, several laboratory tests have been developed.

The simplest is to measure the sedimentation volume of flour suspended in an alkaline medium. High negative correlations between the sediment volume and the flow of the biscuit in the oven are claimed.[3, 4] Two tests which try to follow the conditions given above are the Chopin Alveograph[5] in which a thin sheet of dough is blown into a bubble by a controlled flow of air until the bubble ruptures and the Halton Extensimeter[6] in which the force required to stretch a dough and the degree of extension when the dough breaks are measured. The latter instrument has shown promise in selecting flours for the semi-sweet hard dough biscuit (e.g., Marie or Thin Arrowroot).[7] Various types of the well-known Brabender instruments are also used; mainly for strong flours.

In general a test should aim at a determination of the characteristics which are most vital in the manufacturing process employed. These characteristics vary from one type of biscuit to another. Thus it is a common experience that a flour which is suitable for, say, a semi-sweet biscuit may be unsuitable for a sweet kind such as ginger nut.

Fats

Fat is used in biscuits to remove hardness and to improve the shortness of the biscuits—that is to lower the forces necessary to break and crush the biscuits.

A variety of fats, mainly vegetable, are used (coconut oil, palm oil, hydrogenated groundnut, rapeseed and cottonseed oils, palm kernel oil and hydrogenated whale oil are among the main ones) and these should be bland in smell and taste. Butter and oleo oil are also used and the natural flavourings of these fats are valuable in the finished product.

In most biscuits, particularly sweet kinds, it is necessary that the fat should be soft enough to disperse easily in the dough during mixing and yet remain solid. If the fat melts, the dough may become unworkable on the machine. In semi-sweet kinds the fat will largely melt because of the mixing temperature but should not become hard when the dough is cooled on the rollers. In puff kinds, however, the fat is chilled before use because here it is
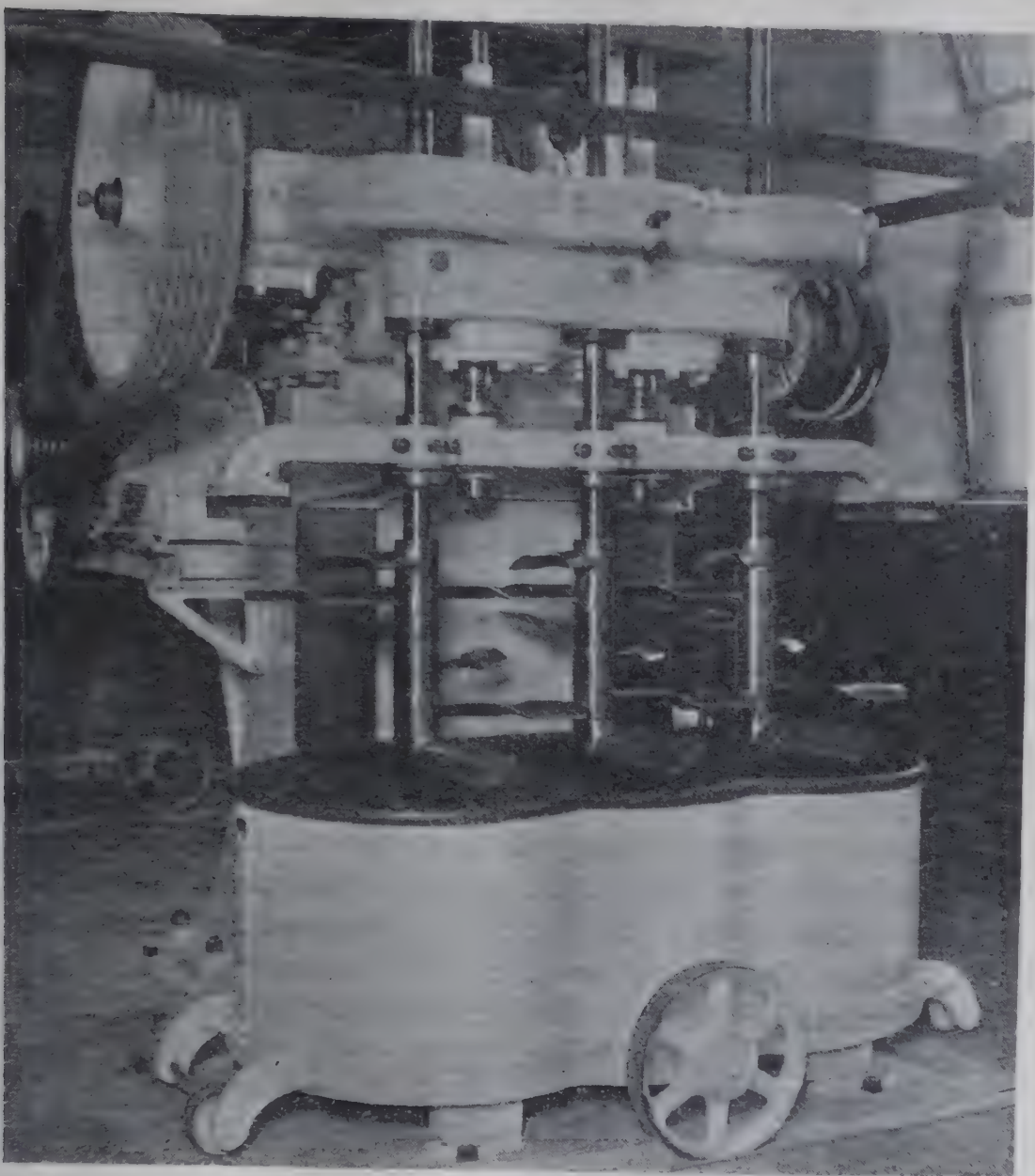
FIG. 1.—A VERTICAL SPINDLE TUB MIXER.
(W. & R. Jacob & Co. (Liverpool), Ltd.

necessary that the fat (incorporated separately into a flour-water dough) should separate the seventy odd layers of dough during the preliminary manipulation and rolling and should not be squeezed out of the side of the layers. At the same time, the fat, when chilled, should not be so hard that it breaks through the layers of dough.

If the fat contains some high melting triglycerides it will be necessary to texturise it, that is to cool the liquid fat very rapidly so that only small crystals are formed. If this is not done the high melting components will, on standing, crystallise first and

form large crystals which will not disperse in the dough. Tex-
turisation also leads to a fat whose consistency varies fairly slowly
with temperature.

Physical tests to determine the hardness and rate of melting
are useful besides the normal chemical and organoleptic tests.
Various penetrometer tests are in use, and a useful method devised
by Williams[8] allows the rate of melting to be found. In this
method the variation of density of the fat with temperature is
found by weighing the fat in a bell submerged in water. The rate
of change of density is greater the smaller the temperature range
over which the fat melts.

Biscuits may be kept for several years before they are eaten
and it is therefore essential that rancidity (due to atmospheric
oxidation) be avoided. This means in practice that the linoleic
acid content of the triglycerides should be as low as possible and
that hardened fats should be selectively hydrogenated to accom-
plish this. Butter is somewhat prone to rancidity but it is
interesting to note that partially rancid butter is often preferred
to the fresh fat. Fats are often tested for atmospheric stability by
accelerated oxidation at, say, 60° C. or 100° C. and are followed
by smell and taste and, for an objective valuation, by peroxide
values. (*See* p. 206.) It is preferable, however, to incorporate
the fat into biscuits and to store the biscuits in air-proof tins.
This, besides bringing the fat into the state in which it will be
used (always an admirable idea in any test) allows the natural
anti-oxidants in the flour to exert their effect.

Anti-oxidants act by being preferentially oxidised and thus
delaying the oxidation of the unsaturated fatty acids in the fat.
Several which have been introduced during the last decade or
so are likely, when their use is permitted in this country, to at
least double the shelf life of biscuits. Butylated hydroxy-
anisole is one of the most promising because it is less water
soluble and less inactivated by metal impurities[9] than others.
This means that its activity is retained to a high degree in the fat
after the dough stage.

Another use of anti-oxidants may be to incorporate them in the
wrapping papers in contact with the biscuits when they are packed.
If the temperature of the pack rises so that the fat melts (as might
easily happen in tropical climates), then fat will be absorbed by
the wrapping papers and a small amount of fat will be spread

FIG. 2.—HIGH-SPEED
DOUGH MIXER-TILTING
BOWL.

(*Baker Perkins, Ltd.*)
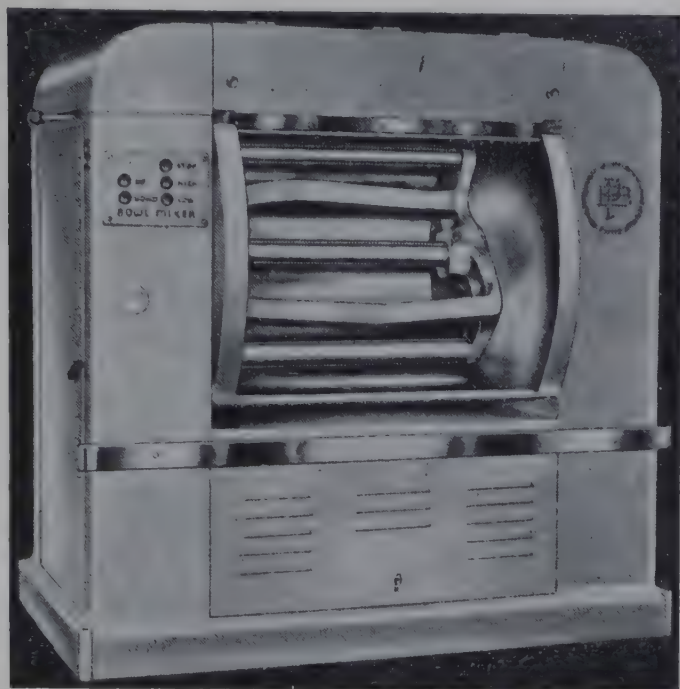
over a large area.   The increased chance of oxidation under these
conditions would be reduced by the presence of anti-oxidants in
the paper.   Certain impurities, mainly metallic, act as pro-
oxidants.   Of these copper is the most important.   0·1 parts per
million has a noticeable effect on fat by itself and a few parts per
million can seriously effect the stability of biscuits.

Other Ingredients

The main *sweetening* agents are sucrose, invert sugar and com-
mercial glucose (glucose plus dextrins).   The monosaccharides
lead to enhanced colour in the baked biscuit because of the
Maillard reaction and polymerisation (particularly under alkaline
conditions) and invert sugar is effective in diminishing checking
in biscuits (*see* p. 110).   Sugars also affect the heat denaturation
of flour proteins (*see* p. 108).

Aerating agents commonly used are ammonium bicarbonate
by itself and sodium bicarbonate with either sodium pyrophos-
phate or calcium hydrogen phosphate.   Ammonium bicarbonate
has the advantage that it leaves no residue and evolves a greater
volume of gas per unit weight than any of the others but, in the
absence of phosphate and sodium ions, it leaves a raw taste in the
biscuits.   Sodium pyrophosphate residues give a burning taste
and excess sodium bicarbonate gives a fine granular texture to
the biscuit (which is desired) but only at the expense of a burning

taste.   Biscuits are normally baked to a pH of about 6.   This gives maximum retention of flavours which are usually of an aldehydic nature.   However, if cinnamon is present a definitely alkaline reaction (pH about 8) is needed if the full flavour is to be kept.

Various safe water-soluble coal tar colours are available but increasing resort is having to be made to natural oil-soluble colours for creams between biscuits as possible health hazards due to the oil-soluble coal tar colours are discovered.

THE BAKING PROCESS

The factors which determine the shape and size of the baked biscuit in the oven are (a) the flow or contraction of the dough as the temperature rises, (b) the rate of loss of moisture and (c) the heat denaturation of the flour proteins.   Starch in this connection is unimportant because very little or no swelling of the starch grains occurs.   Puff types of biscuit are made from strong Canadian flours (Spring wheats) and the stresses imposed on the dough during the rolling process lead to contraction of the biscuits as the temperature rises and the dough becomes less viscous.   This contraction is greater along one axis than along the orthogonal one and, if the baked biscuits are desired to be circular, then the cutter must be oval in shape.   A similar contraction, although not so marked, occurs in cream crackers where the flour is an equal parts mixture of strong Canadian and weak English.

If all-English flour is used then, because of the weaker nature of the proteins, there is a general tendency to flow.   This will be controlled to a large extent by the amount of sugar.   As the percentage of sugar rises so the temperature at which the flour proteins are denatured rises and, therefore, the biscuits will reach a higher temperature and have a greater chance to flow before their shape is fixed.   In fact, in very sweet kinds, such as ginger nuts in which the characteristic surface cracks demand a large amount of flow, the proteins (with the exception of those immediately at the surface of the biscuit) are not denatured at all. In this class of biscuit loss of water is the controlling factor in the amount of flow and steam may be injected into the oven to slow down this loss.   The action of sugars in retarding heat
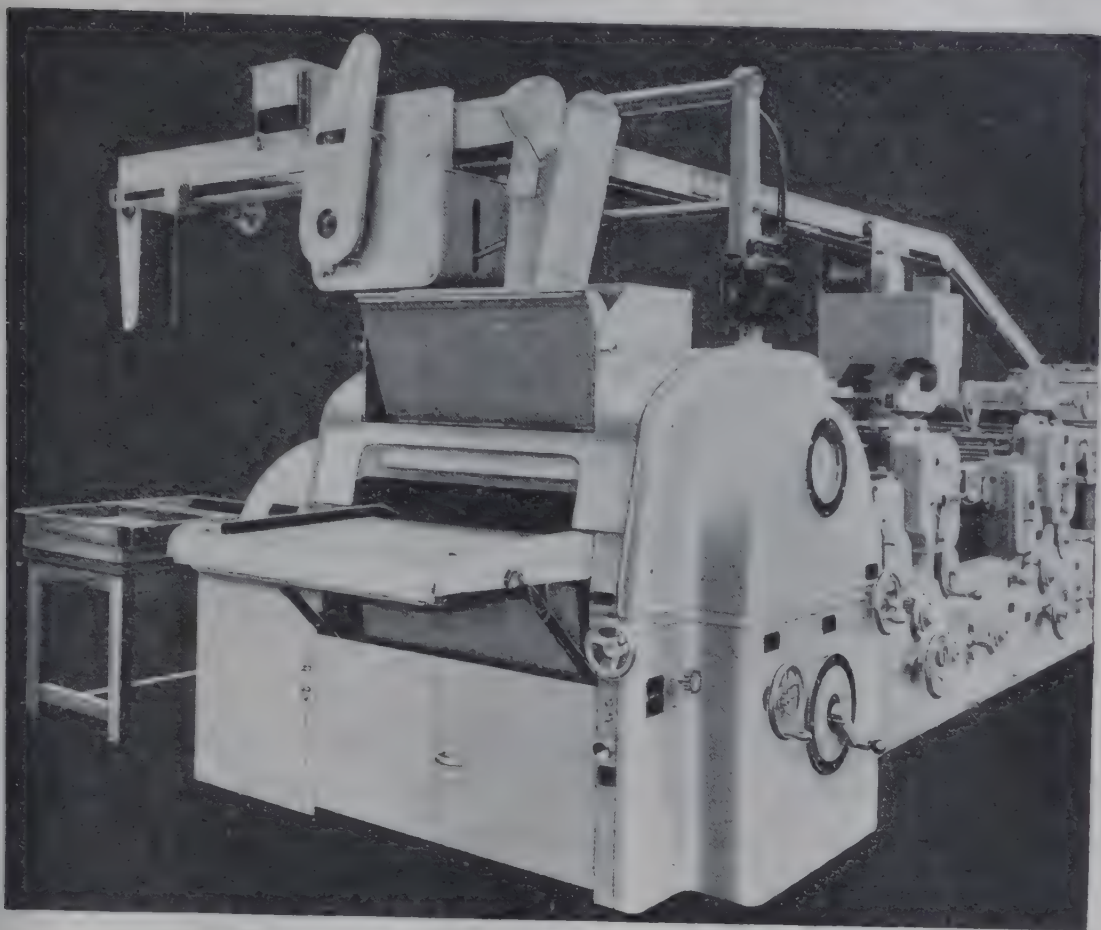
FIG. 3.—HOPPER AND MACHINE FOR SOFT, SWEET DOUGHS.
(Baker Perkins, Ltd.)

denaturation of proteins is vaguely known as peptisation. It presumably occurs through the energy necessary to break the characteristic protein configuration being raised—possibly by the formation of some secondary bonds between protein and sugar.

FAULTS

Checking of Biscuits

It is sometimes found that biscuits after they have been stored a day or so develop cracks which tend to run near the centre of the biscuits but not from edge to edge. This phenomenon is known as checking. A paper by Dunn and Bailey[10] has explained the causes and suggested control measures. The incidence of checking varies with the fat content and the maximum occurs at the level of fat used in semi-sweet hard doughs. The cause lies in stresses set up in the biscuits and these may occur through under-baking, a low relative humidity or too rapid

FIG. 4.—EXPORT WRAPPING DEPARTMENT OF A BISCUIT FACTORY.
(*W. & R. Jacob & Co. (Liverpool), Ltd.*)

cooling. Under-baking results in the outside portion of the biscuit drying out more than the centre portion and, as the biscuit moisture tends to equilibrium, the outer portion will expand and the inner portion contract. A low relative humidity will tend to accentuate the moisture differences; while too rapid cooling makes the biscuits harden before the stresses have been relieved. The inclusion of invert sugar in the recipe tends to reduce checking, possibly by making the biscuit softer and thus more capable of absorbing stresses. In this country under-baking is the main cause of the trouble. Too rapid cooling has been largely eliminated by carrying the biscuits back over the oven while they are cooling.

Bacteriology

Only certain heat resistant bacterial spores but no moulds or vegetative forms of bacteria survive the baking process. Bacillus subtilis, or a similar species, is the most likely to survive but, as biscuits are stored dry (they become inedible if the moisture content rises to 10 per cent), the spores do not germinate. It is only if some moisture rich substance is deliberately deposited on the biscuit that trouble may arise. This is most commonly experienced in chocolate mallows. These confections consist of a biscuit on which is deposited a gelatine-sugar-water foam known as marshmallow. The whole is then covered with chocolate.

Even in this case the percentage of sugar in the biscuit and marshmallow is sufficient to inhibit the growth of bacillus subtilis and the only likely causes of infection are from moulds and yeasts which can grow in a high sugar content medium.   Aspergillus glaucus and several torulopsis are examples.

2.   CAKES

Cakes basically are made from flour, fat, sugar, eggs and water with possible additions of fruit, nuts, milk, colours, flavours and chemical aerating agents.

INGREDIENTS

Flour

A wide range of flours, depending on the type of cake, is used, but weak English flours are probably the most common.

It used to be rare to use more than sixty parts of sugar to one hundred parts of flour but, with the introduction of so-called High Ratio flour, up to one hundred and forty parts of sugar may now be used.   (High Ratio cakes have enhanced eating and keeping properties.)   High Ratio flour is composed of very fine particles and is heavily treated with chlorine containing about 1 per cent nitrosyl chloride.   The chlorine treatment almost completely destroys the formation of flour protein strands in the dough and tends to make the starch more easily gelatinised.   The treatment also lowers the pH of the flour to about 5·0.   Kent Jones and others[11, 12] have shown the importance of particle size. A satisfactory High Ratio flour had no particles larger than 65 microns in diameter (normal flour approximately 28 per cent) and had 55 per cent in the 15 to 25 microns range (normal flour approximately 20 per cent).

Fat

The main actions of fat, apart from acting as a lubricating agent, are to entrap air (creaming) and to emulsify water (see under "Mixing" p. 115).   It is necessary to use fats which tend neither to run to oil nor to be too hard.   This is accomplished by ensuring that the solid triglycerides are in the form of very

small crystals which form a stable matrix and enclose the liquid components. Texturisation, described above, will do this. These kinds of fat are called plastic and are made either by partially hydrogenating a liquid oil or by mixing together a high melting fat with a liquid oil. If the latter is used, the liquid component may go rancid rapidly and a quick sale of the cakes will be necessary. Emulsifying power is increased by including a small percentage of glyceryl mono- and di-stearates which are powerful emulsifying agents. These fats are often called superglycerinated.

Sweetening Agents

Sucrose, commercial glucose and invert sugar are commonly used. Crystalline sucrose enables air to be incorporated into a mix more readily than does pulverised sugar. Probably the crystals, because of their sharp edges, give a stronger mixing effect. Invert sugar, being hygroscopic, tends to slow down the loss of moisture from the cake (a most important characteristic) and, in fact, the more sugar it is possible to incorporate the better from this point of view. This is probably because most of the water will then be present as a syrup which has a lower vapour pressure than water by itself.

Eggs

Eggs in a variety of forms are used. Frozen whole, sugared frozen whole, spray dried whole, sugared spray dried whole, frozen white and dried white are the more important types. Egg white is used in angel cakes and the foaming power of the albumen is important in connection with the size of the finished cake. Whole egg is used in sponges and here again the foaming power is important. Sugar dried whole egg was introduced because it was found that if sucrose or lactose were dissolved in the egg before it was dried then the foaming power of the reconstituted egg was as good as that of fresh or frozen eggs.[13] If sugar was not added then the foaming power would fall by 50 per cent or more.

In types of cakes in which fat is added, there is a connection between the solubility of spray dried whole egg and the eating qualities of the cake but the foaming power is unimportant.[14]

In the latter types of cakes aerating power is provided mainly by the creaming process and by chemical aerating agents.

Liquid egg is an excellent medium for bacterial growth and it is necessary to ship and store frozen egg at $-10°$ C. and to thaw it at temperatures not higher than $15°$ C.   It should then be used as quickly as possible.

Aerating Agents and pH

Ammonium bicarbonate is not used in cakes because all the ammonia might not escape and might taint the finished product. Sodium bicarbonate and either sodium pyrophosphate or calcium hydrogen phosphate are mainly used.   Potassium hydrogen tartrate may also be used but it tends to react with the sodium bicarbonate before the dough is baked.

It is better on the whole to have a pH less than 7 in the finished cake.   This results in a lightening of the colour, an improvement in the keeping properties, more stable emulsions with hydrogenated fats, better retention of flavours and a finer texture. However, if the cake is very acid it may be too tender to handle.

THE MIXING PROCESS

In the majority of cakes in which fat is added, there are two main mixing methods.   The fat is creamed either with the sugar or with an equal weight of flour.   In the fat-sugar batter method the creaming incorporates air and the action of the sugar is purely mechanical.   The original volume should be doubled.   The egg is then added in aliquots and a water in fat emulsion is formed. If the egg is added too rapidly large water drops may be formed which separate out of the emulsion and may cause a complete breakdown.   This is known as curdling.   (The egg should preferably be warmed a little before it is added otherwise the cooling due to the sugar dissolving in the egg may reduce the ability of the egg protein to stabilise the emulsion and curdling may occur.)   The flour should then be mixed in gently so that the foam and emulsion are preserved.   In the fat-flour batter method, the mechanical effect of the flour is not as great as that of sugar

because it is not crystalline and creaming takes considerably longer. The egg and sugar are creamed separately and added and then the rest of the flour is added. No curdling occurs in this method.

THE BAKING PROCESS

When a cake is baked the increase in temperature results in (a) the formation of carbon dioxide from the chemical aerating agents, (b) an increase in water vapour and (c) an expansion of entrapped air. These three factors make the cake rise. The amount of rise and the structure of the cake are controlled by (i) heat denaturation of the egg and flour proteins and (ii) gelatinisation of the starch. The art of cake making lies in obtaining the correct balance between these two opposing sets of forces. Sugars modify the second set by increasing the denaturation temperature of the proteins and by slowing the gelatinisation of the starch. This last is accomplished by the sugar solution reducing the absorption of water by the starch granules by osmotic effects.

Staling

Staling of cake—that is the development of dryness, crumbliness and toughness in eating properties—occurs through two causes, only one of which is partially avoidable. First, the cake may lose moisture to the atmosphere and second, a redistribution of moisture in the cake itself always occurs. Loss of moisture to the atmosphere may be avoided by a water-vapour proof wrapping, but the danger here is that the relative humidity inside the wrapping may become high enough to allow the growth of air borne moulds which have settled on the cake before it is wrapped.

Although a lot of work has been done on the internal redistribution of moisture, particularly in connection with bread, the causes are as yet not fully known. It would seem probable[15] that the amylopectin fraction of the starch becomes gradually insolubilised and tougher as staling develops but the role, if any, of the protein is not known. No method of stopping the development of the second type of staling under normal storage conditions is known, but several compounds have been suggested to keep the crumb from toughening. Glycerine is commonly employed. As it has

a low vapour pressure, it will remain in the cake and probably acts solely as a non-evaporatable liquid.

Bacteriology

As in biscuits, only heat resistant bacterial spores survive the baking process. Bacillus mesentericus is the chief of these and may lead to "ropiness" in the cake. It has been found that acetic acid at about 0·07 per cent on the flour will effectively inhibit the development of the bacillus.

<div align="right">H. N. M.</div>

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CHAPTER 10

CANNING—FRUITS AND VEGETABLES

THE ideal before the canner is to produce an article in which the natural colour, flavour, texture and appearance of the fruits and vegetables are retained indefinitely. The natural vitamin potency must equally be preserved. A combination of scientific research and commercial practice has enabled the canning process to advance considerably towards this ideal. Details of this process are perhaps best described under three heads—the container, the raw materials, and the factory procedure.

THE CONTAINER

This is a very important item. Two alternatives are available —the glass jar and the tin can. The former has several advantages, but the economy of the latter more than offsets these and makes the tin can the popular container.

The so-called "open-top" can has been materially improved in the last decade. It consists of a cylindrical body of tin plate (with or without an internal coating of lacquer) with double-seamed-wheeled-on ends. A hermetic seal at the ends is effected by means of a rubber compound which is previously flowed into the can end flanges, while the double seam of the body joint is secured by a thin film of solder along the outside—i.e., no solder comes into contact with the contents.

The older process of applying the tin by means of hot dipping is being superseded by electrolytic tinning of the continuous mild steel strip, with a corresponding economy in the use of tin.

It has long been known that the complete and uniform coating of the base plate with the film of tin was essential for success, but recent research points to the importance of the composition of the steel base plate.

The Use of Lacquer

Even the best tin plate shows under test, pinholes of bare base metal, and the effect of this on fruit and vegetables gave rise to

the use of lacquer. Suitable gums and gum resins are dissolved in mixed solvents and, prior to making up, the plate is evenly coated with the mixture, which is then dried and hardened by "baking" in hot air ovens. In subsequent handling in making up the can, every care is taken to avoid damage to this protective film.

The prime function of the lacquer is to retard the attack on the tinplate by fruit acids which can dissolve out the iron with the formation of hydrogen in sufficient quantity to give rise to so-called "hydrogen swells." On the other hand, contact with tin results in an unfortunate change in shade of the red (anthocyanin) colouring bodies in fruit from their attractive natural colour to a somewhat objectionable purple. The lacquer film prevents this trouble and for this reason is still necessary with fruits of red shades of colour, but it is not always used for green or yellow fruits.

The lacquer varies in composition according to its use— whether for fruit or vegetables. It must have no effect on either the colour or flavour of the can contents, must be quite insoluble, and must not peel off on prolonged storage. While quite hard, it should not be at all brittle, but sufficiently plastic to avoid cracking and other damage during the manufacture of the cans.

THE RAW MATERIALS

The fruit and vegetables to be canned and, in addition, sugar, salt and water (and in some cases a little fruit acid and, where needed, a trace of suitable artificial colour) cover the range of raw materials.

SUCROSE.—White, refined sucrose is employed, either of cane or beet origin.

SALT.—Salt of high chemical and bacteriological purity only is used in vegetable canning.

DRINKING WATER.—Any good drinking water supply is suitable for canning, provided it is not unduly hard.

THE SUPPLY OF FRUIT AND VEGETABLES

With fruit and vegetables—raw materials for canning—it is important to consider the source of supply, the matter of freshness, the variety and quality, considerations of cleanliness, etc.

Experience has shown that no cannery can thrive on cull fruit or market excess in times of glut. Successful canning calls for fruit or vegetables of selected variety from suitable soil and harvested as to time and manner more from the canners' and less from the farmers' point of view than is normally the case.

The Importance of Freshness

The cannery should be situated in proximity to the district in which the raw material is grown. Freshness is a prime necessity. A popular canning aphorism in America is: "There are just two days when peas are fit for canning—the day they are ready, and the day before." Admittedly, freshness is a comparative term, but the less time allowed for post-picking changes in the fruit etc., before all life action is killed in the canning process, the better the final result. Mould spores are always present with ripening fruit and develop very rapidly after picking. A consignment of ripe strawberries for canning can easily suffer considerable damage from mould development in 20 hours in a ventilated box railway truck during transit, but refrigerated rail and road transport is available, and ripening may also be delayed in a CO_2 controlled atmosphere.

Again, with peas—bacterial development and enzymic action during transit to the cannery, and any delay in handling on arrival, may easily give rise to deterioration in flavour and problems of the "flat-sour" variety. In fact, freshness determines very largely the perfection of flavour, as well as (with fruit especially) firmness of texture—always, of course, assuming that picking has occurred at the correct stage of maturity.

For canning, fruit should be picked just before it reaches the "dessert" stage of ripeness. If picked too soon the cells and tissues are not sufficiently developed and the fruit shrivels in the sugar syrup, and of course the less ripe the fruit the less the flavour. On the other hand, if the point of maximum flavour development be taken as a standard for picking, the texture will be too soft and the fruit will disintegrate in handling and processing. The optimum conditions for canning in relation to maturity must be learned by experience—they vary with different fruits.
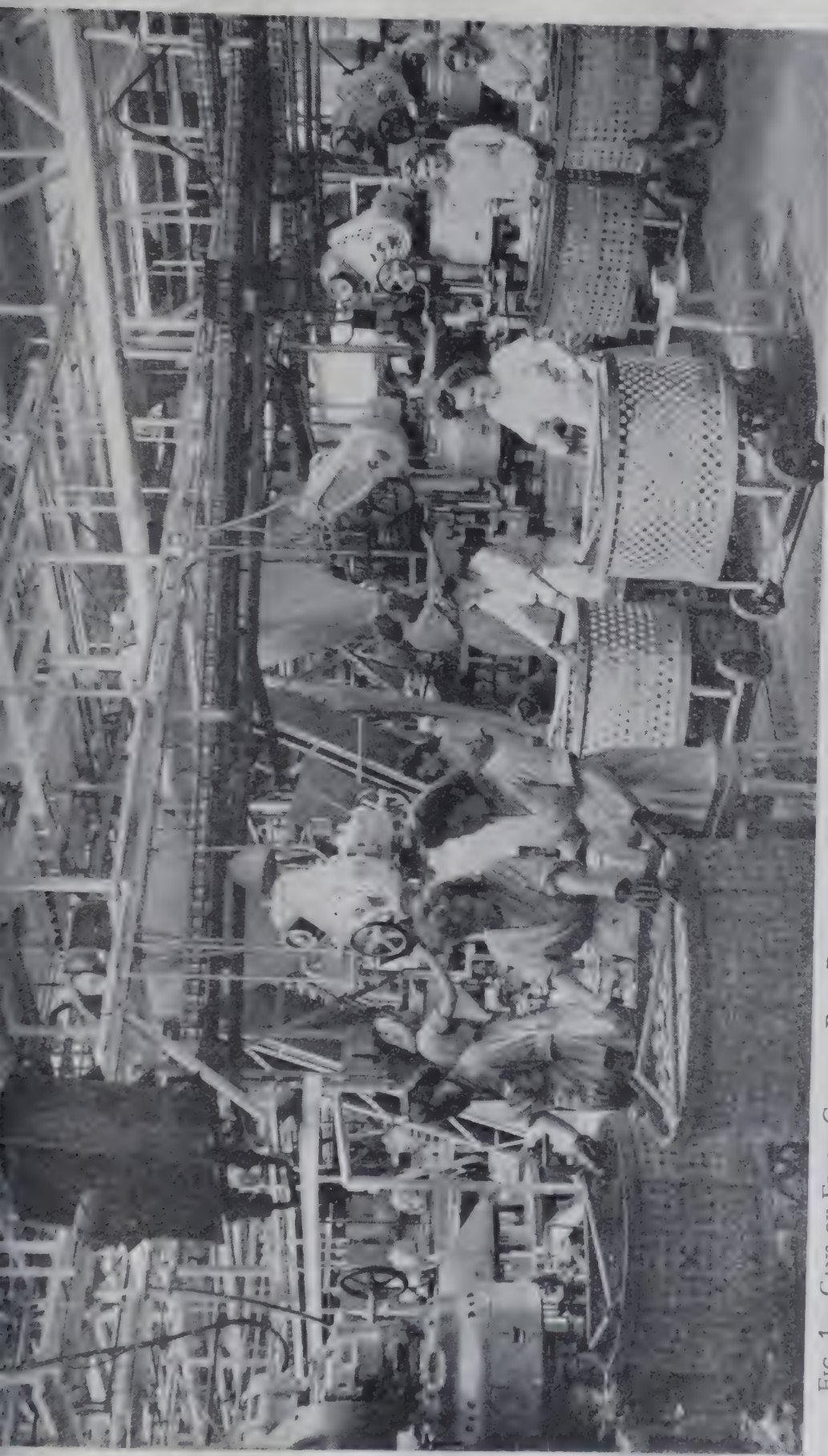
FIG. 1.—CANS OF FRESH GARDEN PEAS READY FOR THE COOKING RETORTS AT CHIVERS' VEGETABLE CANNING FACTORY.

(Chivers & Sons, Ltd.)

Condition of the Fruit

One final point to be remembered is the "condition" of the fruit for canning. Freshness and correct degree of maturity have already been stressed. The fruit may pass on these counts but may still be unfit for canning because of bruises, blemishes, faulty shape, contamination with earth, dirt, leaves, stalks and other extraneous matters.

For these reasons fruit and vegetables pass through a number of important preparation processes prior to the process of canning proper. These include "nubbing," "strigging," "shelling," "snipping," "pitting" as well as hand sorting, washing and grading.

PREPARATION OF FRUIT AND VEGETABLES

Gooseberries

Gooseberries, for example, need to be "topped and tailed," i.e., to have the remnants of the calyx removed from one extremity and the stalk from the other. This is termed in canning circles "nubbing." It may be done by placing the fruit in an open-topped stationary iron cylinder fitted with a revolving bottom of such design that a wave-like rotary motion is imparted to the mass of fruit. The whole interior of the machine is lined with fairly rough-grained carborundum and during use is drenched with a spray of cold water. Alternatively, the fruit may be progressed along revolving abrasive rollers and thereby "topped and tailed" continuously. The abrasive action is such that first the "nubs" are removed from the gooseberries with quite negligible damage to the skin, but if the action is continued the skin is almost entirely removed. The fruit is, of course removed from the machine at the point of maximum loss of tops and tails and minimum skin damage. It is a very satisfactory operation with green firm fruit.

Blackcurrants

Blackcurrants need stalking—this is done on a "strigger," one type of which consists of a slightly inclined tray, the bottom of which is formed by a series of parallel bars through which rotate hooked metal fingers. The tray oscillates, and currants placed at the top end are impelled forward—so passing over the metal

fingers which engage the stalks and drag them through the bars, which are so spaced that the fruit is retained above. Ease of strigging varies with the variety of Blackcurrant and degree of ripeness, but can often be facilitated if the fruit is first frozen hard.

Strawberries and Raspberries

The two most popular soft fruits canned in this country are unfortunately the least readily handled by mechanical means. Raspberries are normally picked "off stalk" but some varieties of Strawberries do not separate easily from calyx and stalk at the time of picking, and need to be "plugged" at the factory. This is almost wholly a hand operation requiring a great deal of labour, although mechanical hullers with a limited degree of efficiency are beginning to be available.

Cherries and Plums

Stones are removed from cherries and certain varieties of plums on the manufacturing scale by "pitting." The fruit falls into holes placed in regular rows in a belt, which places it in position beneath a series of fine metal prongs so spaced that while the fruit is held firm and suffers the minimum of damage, the stone is pushed out through the flesh and skin.

Peas

Peas are generally harvested together with the vine on which they have grown and carted intact to the viner, which may be situated at farm or factory. In some areas mobile viners are in use whereby the peas are harvested and shelled on the field. The exact time of cutting has until recently been decided by the grower or the factory fieldsman from experience, but it has now become a widespread practice to use maturity rating instruments, of which the Tenderometer is an example. Small samples of shelled cut peas from the crop are tested on this machine, which by the force required to effect a shearing action indicates on a dial the maturity rating of the sample. For each variety of pea grown for canning there is an optimum maturity rating corresponding to an ideal condition for canning and a satisfactory crop yield to the grower. Once this stage is reached, the whole of the field is cut as expeditiously as possible.

The pea viner, which consists of a rotating perforated screen

with internal beaters, is loaded with peas on the vine at one end and discharges the spent vine at the other end while the threshed out peas pass through the perforations to the winnower.

Beans

This heading refers to the dwarf french or runner bean picked green and before it has time to develop seeds. No domestic kitchen welcomes the tiresome job of preparing beans for cooking. There are no strings to remove from canning beans (a stringless variety is specially grown) and the tops and tails are removed in "snippers." These consist of revolving, horizontal cylinders fitted with helical baffles and with the walls perforated with many suitably shaped holes. As the beans traverse this cylinder the ends project through these holes and are sliced off with an external oscillating knife suitably attached. The beans are then fed into finger-like hoppers of the slicing machines, the revolving knives of which quickly reduce them to uniform slices.

STAGES IN PROCESSING

All this mechanical treatment needs supplementing with hand sorting. Indeed, there is no substitute for this for the removal of blemished, damaged, under or over-ripe fruit, adhering stalks, leaves, etc.

Washing and Sorting

Wherever possible, fruit for canning should be washed. Strawberries, for example—even from beds that have been well strawed—often carry an appreciable amount of grit and earthy matter from the soil. The amazing amount of sandy sludge taken from a large-scale mechanical strawberry washer at the end of a day's run is very convincing proof of the need for the careful washing of fruit for canning. Pea-washing plant ingeniously removes heavy soil and sand and at the same time light, floating extraneous matter is washed away, and the peas cleansed of gummy and similar substances. Washing machines vary in their design according to the fruit or vegetable they are intended to handle. Some are built on the principle of a belt on which the fruit is carried beneath a series of water sprays, in some the fruit revolves on a rubber or fibre mat under water, and some again employ a revolving brush idea to assist a water spray.

Root vegetables are often washed and peeled in one operation. The same machine used for gooseberry nubbing may also be used to wash and peel potatoes and carrots, but other methods are in use employing lye, flame or high pressure steam. One advantage of the last three is the ease with which eyes are removed from potatoes without rubbing away the flesh.

Grading

It is obviously desirable to have fruit of uniform size in a given container, and as British fruits and vegetables vary considerably in dimensions, "grading" or sorting according to size is a necessary operation. With strawberries and raspberries this must be done by hand, but in the case of gooseberries, cherries, plums, apples, carrots, peas, etc., mechanical graders are used. These again vary very widely in design.

Plums are graded on machines fitted with wooden slats, the openings between which vary in width as the slats progress along the machine. The falling fruit is received into chutes beneath the slats. On another machine, a device is fitted to give a spinning motion to individual plums to compensate for their irregular shape and so ensure uniform grading. Apart from this device, it will readily be seen that a plum lying with its longitudinal axis at right angles to the opening would pass to a larger grade than the same-sized fruit which happened to be with its transverse axis across the opening.

Peas are comparatively easy to grade owing to their spherical form. A revolving cylinder perforated with holes the size of which increases in regular stages from the inlet to the discharge end, and through which the peas pass, is a simple but effective form of pea grader.

Filling into Cans or Bottles

After these many preliminary operations the fruit is now ready for filling into cans or bottles. In the main, this is done by hand. Mechanical filling must reckon with the necessity of filling the container to its utmost capacity with the fruit or vegetable—consistent with the avoidance of crushing. With peas this is possible, and it is general practice to fill mechanically, but the irregular shape of most fruits and vegetables calls for hand work to get a full container. This is especially so with bottles, where the natural

FIG. 2.—FILLING THE CANS WITH FRUIT.

The cans are filled with fruit and placed on the conveyor which carries them along to the syrup machine.

(*Chivers & Sons, Ltd.*)

shrinkage which occurs in the cooking process, and which is so easily apparent in a glass container, gives an impression of under-filling.   To offset this, fruit for bottling and for so-called "solid pack" cans is "blanched" before filling.   Many vegetables, too, are "blanched" before filling into cans.

" Blanching "

"Blanching" is simply a partial pre-cooking.   The fruit is plunged for a given time—from half to, say, five minutes, according to variety—into water at from 180° F. to 200° F., and then immediately cooled by immersion in cold water.   The object is to soften the texture and so enable a greater weight to be pressed into the container without damage to the individual fruit.

With vegetables, "blanching" has a variety of functions. Shrinking, removal of air, and in some cases, as with peas, the removal of undesirable flavouring substances.
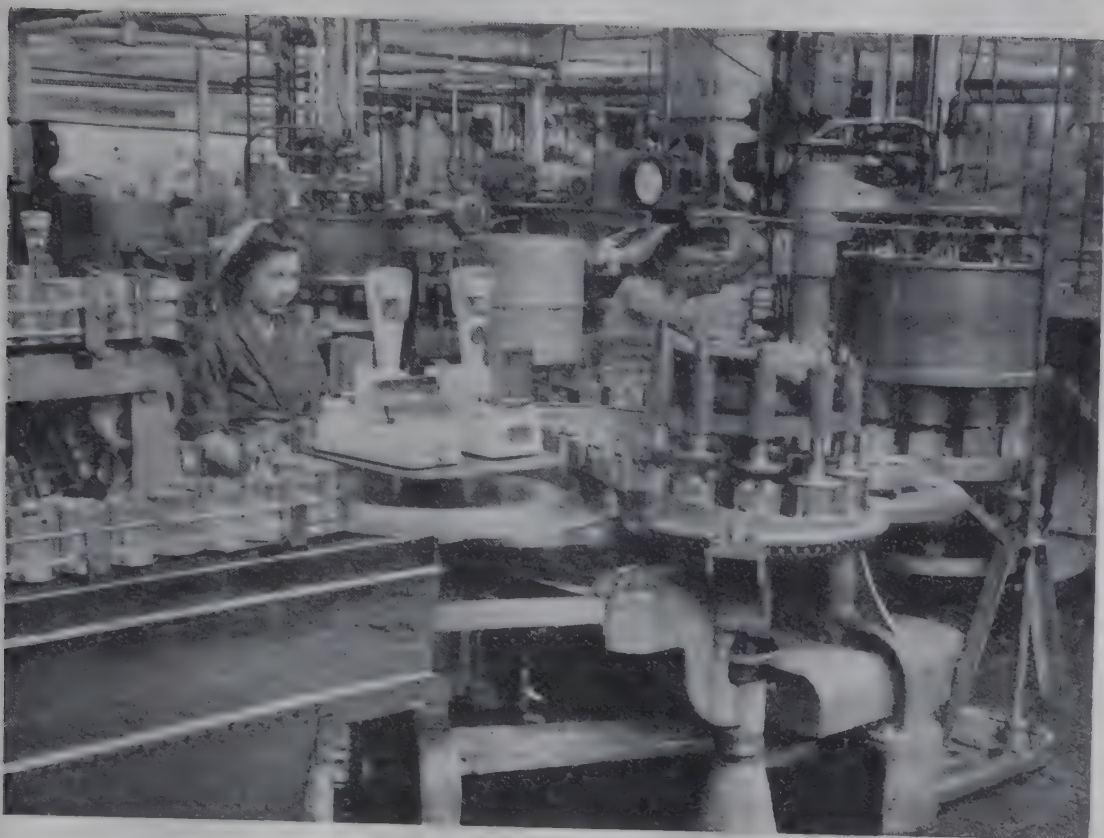
FIG. 3.—WEIGHT TESTS ARE MADE AS THE CANS PASS TO THE MACHINES
WHICH ADD THE SYRUP AND SEAL ON THE LIDS.

(Chivers & Sons, Ltd.)

Addition of Liquor or Syrup

The liquor, or syrup, is next added—usually mechanically.   It
is always added as hot as possible.   It is also important that any
liquid added should be clear and bright—it is usually filtered
before use.   Any colouring matter needed for a given pack is
dissolved in and added with this liquid. The amount of liquid
added is regulated so that in the finished can there is from $\frac{3}{16}$ in.
to $\frac{5}{16}$ in. "headspace," i.e., space void of liquid or fruit.

Fruits (especially in large cans) designed for use in tarts and
cooking generally are usually packed in water. The popular
pack is, of course, in sugar syrup so that on opening the can the
contents are ready for immediate consumption without any
addition or further cooking. Great care is taken to keep the
sugar concentration of the syrup constant and at a fixed or standard
strength.   This is arrived at mainly by considerations of flavour,
which are influenced by the acid and general character of the fruit
used.

It is to be remembered that in the finished can a state of equilibrium will slowly occur within and without the fruit by osmosis. The sugar syrup is diluted with the juice passing out of the fruit cells, and the concentration of the soluble cell contents rises as sugar enters. The stronger the syrup that is used, the greater the tendency for the fruit to shrivel (which should be avoided) and the greater the final shrinkage. At the same time, sugar has the effect of making the fruit texture firmer and of imparting a desirable brightness and sparkle to the appearance of the fruit. It is to its homogeneity and to the controlled effect of sugar on texture that canned fruit owes its superior flavour and palate effect—a result which direct cooking can never approach.

Vegetables are usually packed in brine, that is to say water to which a small addition of salt and sometimes also of sugar has been made.

The Exhauster or Preheater

While some heat units have been introduced by the hot liquor, more are needed. "Exhausting" is therefore the next process. The lid is placed loosely in position and the can enters the "exhauster," which is a lidded tank fitted with revolving wheels, or conveyor chains, or other means to cause the cans to progress forward through water or steam kept at a minimum of 200° F. The cans are immersed in the water to at least 75 per cent of their height. The speed of the conveyor or wheels is regulated to allow a definite time for exhausting. This varies from 1 to 10 minutes, according to the size of the can and the variety of fruit.

Like "blanching," "exhausting" serves at least a triple purpose:

1. It helps to remove air from the fruit or vegetable tissues.
2. It ensures a higher final vacuum in the can.
3. It assists the cooking and sterilising process.

"Steam flow closure" is an alternative method of achieving the same objective whereby a puff of steam is injected into the headspace immediately before sealing on the lid, thus displacing the air and condensing to leave a vacuum.

Vacuum

A good "exhaust" raises the internal temperature, and air and other gases in the headspace are largely displaced by expansion

and replaced by steam. Sealed in this condition a can will show good vacuum on cooling. Under proper canning conditions a vacuum of 10 to 14 in. of mercury should result. The production (within limits) of hydrogen in a can is no detriment to the contents, but nevertheless ranks as spoilage owing to bulged can ends. A good vacuum means, therefore, among other things, that a fair production of hydrogen may occur without bulged ends, i.e., without the commercially so-called "half-blown" effect.

The raising of the temperature of the can contents during "exhausting" helps the attainment of the thermal death point of yeasts, bacteria, etc., under canning conditions, earlier in the cooking process and so helps to ensure the sterility of the pack.

Sealing the Lid

The "exhausted" can now passes to the "double seamer"—which machine seals the lid securely to the body of the can. It is most important this operation should be mechanically accurate and perfect. The flange of the can body and the outer rim of the lid—between which is sandwiched a gasket of rubber latex—are given a double fold and then squeezed tightly together. This is done by causing the flange edges to impinge on wheels of suitable contour revolving at high speed, simultaneously revolving around the can.

Faulty adjustment of the wheels or rolls of this machine results, on the one hand, in fracture or cutting of the metal, and on the other, in a joint insufficiently tight. Both faults mean spoilage. Expert knowledge is required to detect the minute faults in double seaming which, though apparently quite air-tight on test, yet under the strain of cooking and cooling admit a trace of air or water and with it micro-organisms, with consequent spoilage.

COOKING OR "PROCESSING"

Cooking, or "processing" in canning parlance, are terms for the next stage of cannery practice, the meaning of which is self-evident.

From the consumer's standpoint the fruit is cooked. From the canner's it is "processed," i.e., cooked and sterilised. These are the two main functions of this department of the cannery.

The fruit or vegetable must be whole and cooked to the optimum point of tender firmness. Such a result needs rigidly controlled conditions of time and temperature—especially when the factor of sterility is added. Fortunately, with the pH values incidental to fruits in sugar, most yeasts, moulds and bacteria are killed within conditions compatible with good cooking. With vegetables, which have a higher pH than fruit, pressure cooking, with a consequent higher temperature, is essential to ensure commercial sterility. For this reason the home canning of vegetables is dangerous and to be deprecated. American experience has shown the serious danger of botulism in home-canned vegetables. The cellular structure of the vegetables being firmer than that of fruit, this additional cooking is no detriment.

Bacteriological Checks Are Advisable

It must not be assumed from the above that bacterial troubles in canning are unknown. No can of fruit should leave the factory until sufficient time has elapsed to ensure freedom from fermentation due to yeast cells which may have survived. A laboratory bacteriological check on test cans of vegetables is also a wise item of factory routine. Resistant moulds, such as Byssochlamys fulva, too, are known, and "flat sours" in canned peas are produced by thermophilic bacteria which have escaped death in the cooking process.

Types of Cookers

Obviously, cookers for fruit must be quite different in design to those for vegetables. There are two main types of the former—in one the cans are propelled through a heated chamber, without themselves revolving, and in the other the cans themselves revolve during the entire cooking process. The advantage of the latter is at once apparent when it is remembered that the more uniformly the contents of the can are heated, the better the result. The quantity of fruit (as, for example, close-pack apples) in the can hinders and sometimes prevents convection currents, and the penetration of heat by conduction only is a slow process. When, however, the can contents are gently agitated, quicker and more uniform heating results. At the same time care must be exercised not to carry the agitation too far, particularly during the later stages of cooking, when the fruit is more tender and therefore
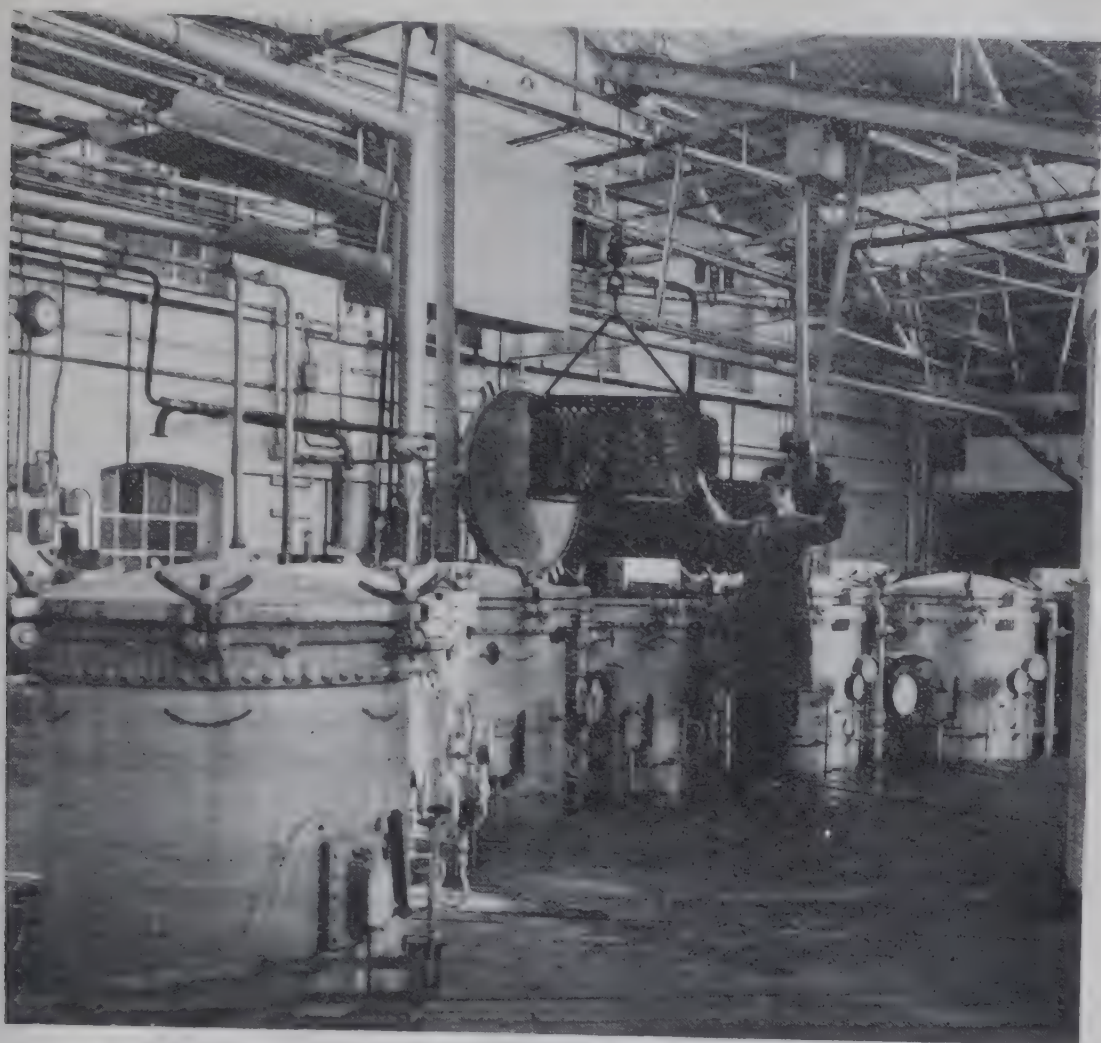
Fig. 4.—Loading Canned Vegetables into Pressure Cooking Retorts at Messrs. Chivers Vegetable Canning Factory at Huntingdon.

liable to disintegrate. To ensure sterility, the *centre* of the can must reach a given temperature, but at the same time the temperature of the fruit adjacent to the sides of the can must not rise unduly high or over-cooking may result.

In practice it is not possible to obtain ideal conditions from each point of view, but a satisfactory compromise is readily achieved.

The Rotary Cooker

One type of rotary cooker for fruit consists of an outer cylindrical jacket surrounding a revolving metal cage into which the cans are fed at the loading end. The design of the cage is such that the cans follow a helical course towards the outlet end of the cooker, themselves rotating in the process. Upon entering, the cans are immediately immersed in water maintained thermostatically at the required temperature, and removing facilities are

placed at intervals along the cooker, so that the time a given can is immersed in the hot water may be determined at will without altering the speed at which the cooker rotates. Further modifications of time of cooking are controlled by varying the speed of rotation.

Cooking Times and Temperatures

Cooking times and temperatures are fixed by a variety of factors, among which may be mentioned the size of the can, the kind of fruit, and the degree of ripeness.

For practical working, the exact conditions must be fixed by an actual test, and correct cooking maintained by opening test cans at intervals during the day's run. The temperature attained in the centre of the can must also be regularly checked. One method of doing this is to fix a small thermometer, just covering the required range, and fitted with a maximum temperature indicator, in the centre of a test can, suitably marked for identification purposes.

Vegetables may be batch cooked in autoclaves under pressure. The closed cans are filled into perforated metal crates which are placed by mechanical means into cylindrical steel retorts, where they are heated by steam to at least 240° F. for a period of time which varies according to the type of pack and the size of the can. Continuous pressure cookers of various types have been designed and are particularly useful in a mass production line where large quantities of the same type of vegetable are being processed and where the cooking time remains fairly constant. Many of these continuous cookers are operated at temperatures up to 260° F. which represents about the maximum which the can will tolerate.

The Cooling Process

Cooling is the next process. It is important that when the contents of the can are cooked and sterile, the temperature should be lowered as quickly as possible to at least 100° F. Unless sufficient cooling is promptly carried out, not only does the cooking process continue to some extent, but so-called "stack burn" may occur, for imperfectly cooled cans stored in large stacks retain their residual heat for many hours, and even days. Again, faulty cooling accentuates half-blown or hydrogen swell troubles.

Coolers are of varying design, but are often similar in con-

struction to cookers; indeed, vegetable cooking retorts are often fitted with a cold water supply and used as coolers. Care must be taken to avoid straining the cans by too sudden changes from pressure to vacuum. Such strains in a mild form sometimes cause minute temporary openings through which a little cooling water gains access to the can, and this introduces infection with micro-organisms. The bacterial purity of cooling water is therefore important. One method of easing the strain is to maintain an external air pressure on the cans in the cooler, thereby compensating for the drop in steam pressure.

Air cooling in conjunction with a cold water spray is effective in removing heat from cans by means of the latent heat of evaporation. This principle is employed in commercial work in the "tunnel" system of cooling cans.

Over-cooling involves the danger of wet cans being placed in store and paves the way for rusting troubles.

The cooled cans are now ready for the store. This should be a cool building, so ventilated that the humidity of the air in contact with the cans should be low enough to prevent the formation of rust, irrespective of outside atmospheric conditions.

PHYSICAL AND CHEMICAL CONTROL TESTS

The canning and bottling industry involves highly technical operations. It therefore calls for numerous control tests both physical and chemical. The reader is referred to technical books on canning for a complete range of suitable tests, and, indeed, beyond these it is desirable in each cannery to devise special checks on plant operations which must of necessity be suited to the needs of the particular occasion.

Concentration of Sugar Syrup

As pointed out earlier in this article, the concentration of the sugar syrup in which the fruit is canned and the amount of acid present have an important bearing on the flavour and quality of the final pack. These two factors lend themselves readily to analytical control, and routine tests are regularly carried out in all well-appointed factories.

(i) SIMPLE HYDROMETER TEST (p. 175).—It is customary for

the foreman approximately to adjust the strength of his sugar syrup with a hydrometer.   As this is done on the boiling syrup, the final adjustment must be made by the laboratory.

(ii) THE REFRACTOMETER TEST (p. 174).—To do this, a sample of the syrup is cooled to a standard temperature, and the total soluble solids ascertained by measurement of its refractive index. The cooling is conveniently effected by pouring the syrup into a funnel commanding a vertical spiral condenser.   The bore of the condenser tube should be sufficiently restricted to ensure the rate of flow being consistent with the necessary degree of cooling. By employing a refractometer with a scale graduated for per-centages of sugar, these tests can be performed very quickly by a junior assistant.   There is, therefore, no excuse for syrup of incorrect strength being used in any modern canning process.

Weight and Volume

It is readily obvious that the weight and therefore volume of fruit in a given can is complementary to the amount of syrup that can subsequently be added, and that a decrease or increase of the former reacts very considerably upon the proportion of sugar present in the final pack, and consequently upon the flavour.   It is not possible in commercial practice to weight the contents of every individual can, but apart from mechanical aids supplied to operatives who actually place the fruit in the cans, a laboratory check on the fruit in sample cans withdrawn from the canning line immediately prior to syruping assists in maintaining a standard pack.   In the same way, similar control should be exercised in the times and temperatures of exhausting, cooking, cooling etc.

" Cut Out " Tests

A further series of tests is carried out on samples of the finished pack.   These are known as "cut out" tests.   It is not easy to obtain representative samples of the day's run, as fluctuations in the operating of the canning line may easily occur between the taking of the samples, and so pass unnoticed.   The frequency with which such samples should be taken must, therefore, be governed to some extent by local circumstances, but for usual control purposes it should be sufficient if duplicate sample cans are withdrawn from the discharge end of each canning line for, say, every 5,000 cans produced.   One can is tested immediately,

and the second is retained under normal storage conditions for a given period (usually about three months), and the same series of tests then applied.

Degree of Vacuum

The degree of vacuum within the can is a prime indication of correct canning practice. It is, therefore, checked on each test can. A special vacuum gauge is used for this purpose. It consists of a delicate gauge of the orthodox circular type, the connecting tube of which terminates in a tubular needle point surrounded by a pad of pliable rubber. The whole is so dimensioned that, when the point is pressed into the lid of a can, the rubber forms an effective seal around the resulting opening, ensuring an accurate registration on the gauge of the vacuum within the can.

The ends and sides of the larger size cans are necessarily more flexible than the smaller ones. This flexibility affects the vacuum within the can, and allowance must be made for this in interpreting the results of a vacuum test. The smaller-sized cans when first packed should show a vacuum of at least 15 in., but with the larger cans it seldom rises above 8 to 10 in. Vacuum tests on cans three months old will show slightly less than the above figures, but any indication after this period of a definite and obvious fall in the degree of vacuum should be regarded as a warning of either internal gas generation or a leaky container—both faults occasioning spoilage and rendering the pack unsaleable.

Headspace

In dealing with practical canning instructions in the paragraph (p. 127) describing the addition of sugar syrup to the can, mention was made of the "headspace." With vegetables, sufficient "headspace" is essential, as otherwise very serious deterioration and even buckling of the side seam of the can will take place during cooking and cooling operations. Again, with fruit the presence of sufficient "headspace" is important for the maintenance of the original vacuum during the normal period between packing and the final consumption. Hydrogen is generated very slowly as soon as the can is packed, and insufficient "headspace" with a given volume of hydrogen production obviously lowers the vacuum more quickly than a "headspace" of normal proportions,

and so tends to give rise to "half-blown" effects sooner than is desirable.

A measurement of the actual "headspace" is therefore carried out on each test can. This is done by placing a straight edge across the flanges of the opened can and measuring the distance between this and the surface of the liquid. An appropriate subtraction from this figure must be made to allow for the depth of the flange, and the result is usually expressed in sixteenths of an inch.

Appearance and Flavour

Other quality factors which need testing are flavour and sweetness, wholeness of fruit, uniformity of size grading and freedom from blemishes and extraneous matter. In Great Britain an inspection check of the cannery's own findings may be obtained by submitting sample cans to the Fruit & Vegetable Canning Research Association, which maintains a research establishment at Chipping Campden in Gloucestershire. This Research Association, supported jointly by industrial subscriptions and Government grant, acts in a general advisory manner towards its canner members, as well as carrying out research of a more fundamental and long term nature.

Purity and Nutritive Value

There are in existence under the Food and Drugs Act certain statutory regulations governing the use of adulterants, preservatives and colouring matter in foods, which must be observed by the canners of English fruits and vegetables, while the requirements of the Factories Act deal with matters of a more general nature, such a cleanliness of premises and general hygiene. Chemical and bacteriological cleanliness, however, can only be checked by qualified technical staff and mention may therefore be made of the most important standards of this type which every fruit and vegetable canning factory should attain.

Bacteriological contamination of canned vegetables may occur if sufficient washing is not given to the raw vegetables or if the final sterilisation process is not efficiently controlled. The importance of washing has already been stressed and as regards sterilisation, much work on the subject had already been carried out in the United States before large scale vegetable canning was developed in this country, as a result of which safe cooking

temperatures were laid down for non-acid canned foods. Account was taken of can sizes, initial temperatures of can contents, extent of pre-heating, etc., and very precise data obtained on which was based specific recommendations for all the common vegetables and can sizes.

As previously mentioned, the cooking of non-acid foods must be carried out at temperatures well above the boiling point of water and it has been stated that continuous boiling for over 10 hours at 212° F. would be necessary to secure sterilising efficiency equivalent to the recommended pressure cooking times and temperatures, and this would obviously be impracticable. It is safe to say that the United States specifications are observed by all canners in this country, as a result of which, canned foods are claimed to be less liable than ordinary foods to be a source of food poisoning.

With regard to the nutritive value of canned fruits and vegetables, there are two stages of the process in which loss might occur, one in the blanching process and the other in the final cooking. During blanching, water-soluble nutrients, such as natural sugars, mineral salts, and some of the vitamins may be leached out into the blanching water. Losses from this cause can however be minimised by careful process control, and should not be serious. In the cooking process there would be appreciable destruction of heat-sensitive vitamins if the air in the can had not been previously expelled during the pre-heating or exhausting stage, but in the reduced air content of the can little loss occurs.

Some extraction of the water-soluble constituents occurs into the brine or syrup during the process and it is for this reason that emphasis is laid upon the importance of using the liquid as well as the solid contents of the can.

On the credit side, canning factories normally obtain supplies direct from the growers and fruits and vegetables for canning are not subject to the unavoidable delays attached to distributing and marketing of fresh produce. On balance, it can be said, and such a statement backed by scientific evidence, that qualitatively and quantitatively, canned fruits and vegetables compare favourably in nutritive value with fresh foods as normally produced and prepared in the home.   W. E. R.
C. M. M.

K

CHAPTER 11

REFRIGERATION

IT is part of age-old human experience that foods remain in fresh condition longer in cool than in warm weather and, in countries with long, hard winters, it has always been common practice to allow meat and fish to freeze and remain frozen in order to preserve it. Hence it is not surprising that one of the first applications of mechanical refrigeration was to cool perishable foods to prolong their storage life.

The forms of deterioration to which foods are subject at ordinary temperatures and which can be either retarded by refrigeration or are of importance in relation to it are due to the following causes:

1. MICRO-ORGANISMS.—The growth and activity of micro-organisms (moulds, yeasts and bacteria). These can affect all foods containing sufficient moisture for their development, giving rise to mustiness, fermentation, rotting and putrefaction, together with unsightliness and discoloration due to visible growths.

2. OTHER BIOLOGICAL ACTIVITY.—The continuation of biological activity in fruits, vegetables, eggs and other products which are still alive although cut off from their source and from further nourishment. Such activity normally leads to over-ripeness and other manifestations of senescence.

3. AUTOLYTIC CHANGES.—Autolytic changes due to enzymic activity can result in undesirable changes in texture, discoloration and the development of off-flavours (enzymic rancidity).

4. DIRECT OXIDATION.—This can also cause discoloration and the development of off-flavours (oxidative rancidity), particularly in fatty foods.

5. INFESTATION BY INSECTS.—Insects usually become sluggish around 50° F. and begin to die off around 40° F. and need not be further considered.

6. EVAPORATION.—Drying out and wilting due to evaporation.

Other forms of spoilage, e.g., by rodents, contamination by dirt and chemicals, the absorption of foreign odours and mechanical damage, taken altogether, are probably less serious in refrigerated storage than in any other form of warehousing and will not be considered in any detail here.

PHYSICAL, CHEMICAL AND BIOLOGICAL CHANGES

The first four causes of spoilage all involve chemical activity in the stored material and it is one of the basic facts of chemistry that the rate at which any given chemical change proceeds is approximately halved by a fall in temperature of 10° C. Similarly the rate of evaporation of moisture from stored material, other things being equal, is also directly related to temperature, and it is on these two facts that the efficacy of refrigeration for preserving foods rests.

Food, however, is usually composed of complex biological material and this complicates what would otherwise be a straightforward problem, particularly when the food is in the living state and has to be as far as possible preserved unimpaired in that state. Thus, as we shall see later, there are fruits and vegetables which never mature properly after having been stored well above freezing but below certain temperatures which are critical in each case. Instead, they may undergo abnormal or even pathological changes. Fortunately, however, there are many which are not so affected and since, for controlling micro-organisms, the lower the temperature the better, down to the limit of their activity, fruits and vegetables in this latter class should be stored as near freezing as possible.

In frozen storage we are faced with the profound physical effect of actual freezing, not only on living material, which it kills, but also on the flesh of animals which have been slaughtered. In fact, the difference between the problems which confront us in storage above freezing and in storage below freezing is so great that we shall consider them under separate headings. Commodities thus fall into two main groups, (i) living fruits and vegetables and (ii) animal and fish carcasses with, possibly, a third heading (iii) for processed and prepared foods. In the first group, storage is necessarily above freezing if they are to be

consumed alive; in the second and third it may be either above or below according to circumstances and requirements.

As regards drying out and wilting, although these are reduced in rate by cooling, they can be serious to a degree depending on the nature of the material and the length of the storage period. Thus, with salads in short-term storage above freezing, crispness is all-important and, in frozen storage, surface drying or "freezer-burn" can spoil the colour and appearance and, consequently, the acceptability of foods which have been stored for long periods.

Evaporation and Condensation

Control of evaporation at any given temperature is largely a matter of humidity control and, generally, it is impracticable to control the atmosphere of a store containing material of high water content very strictly, although conditions of temperature and humidity should be made as even as possible inside a store by means of fans for air circulation and by proper arrangement of the contents. Where necessary, as with eggs and vegetables (*see* below), humidity can be controlled to some extent by adjusting the area of the cooling surfaces (pipes etc.) and the temperature of the refrigerant, thus altering the temperature-gradient between the cooling surfaces and the stored materials and, consequently, the amount of moisture from the materials condensing on the cooling surfaces.

A further point which has general as well as special significance is the question of the condensation which is bound to occur when material is removed from a cold store. In some cases this may retard wilting and so be beneficial rather than otherwise; in others it may cause loss of bloom and stimulate the growth of micro-organisms which are still alive and potentially active. On the whole, foods which have been cold-stored will not keep as long as fresh foods and should be dealt with promptly.

ORDINARY COOL STORAGE

Fruits

Fidler[1] has given the following list of fruits which can be stored in what he calls "entrepôt cool storage" up to 10 days at 32° F. to 34° F.: American apples, grapes, pears (before they have begun to "turn"), stone fruits (plums, peaches, apricots, cherries), cantaloupes (for short periods of 7 to 10 days). For storage at 40° F. to 45° F. his list includes English apples, cran-

berries, cucumbers, grapefruit and oranges, limes, ripe pineapples, honey-dew melons and water melons (7 to 10 days).

In a store given over entirely to English apples the temperature may be reduced to 37° F. or 38° F. and, under these conditions, good keepers like Bramley Seedling, freshly picked at the right stage of maturity, can be kept about 2 months longer than in stores without temperature control. A few varieties can be kept at 34° F. but most of the more important varieties of English apples tend to undergo an internal physiological breakdown of the tissue (browning) below 38° F.

Another form of damage to which apples are prone when confined in a store is a skin injury known as "scald." This takes the form of brown patches on the skin with browning and softening of the underlying tissue, and is due to the accumulation of volatile products (such as ethylene) given off from the fruit during ripening. It can be controlled to some extent by ventilation or, better still, by wrapping the fruit in paper impregnated with mineral oil which preferentially absorbs the volatiles.

Although plums are mentioned on the above list, this is probably only intended to apply to medium-ripe plums. The flesh of unripe plums of some of the earlier varieties, like Victorias, becomes jelly-like and brown around the stone at 32° F. to 34° F. At 40° F. to 45° F. they ripen normally. But even at 45° F., William bon Chretien pears from S. Africa and Australia have shown internal low-temperature injury after only one week's storage. Tomatoes rapidly develop watery spots and lemons darken between the segments if stored below 50° F. to 58° F., while for bananas there is a critical temperature near 54° F. below which the skins become a dull khaki colour and the flesh hard, insipid and astringent on being transferred to a temperature of 65° F. for ripening.

If they are firm and clean initially, strawberries can be kept 2 or 3 days, blackberries, loganberries and raspberries about a week and blackcurrants up to a fortnight, but soft fruits in general tend to lose their "bloom" through condensation after removal from store and are best either consumed or processed promptly.

Vegetables

All green vegetables, salads (preferably sprinkled with ice) and root vegetables, except new potatoes, can be stored for

shorter or longer periods according to the nature and condition of the product. For new potatoes, 45° F. is better than 40° F. as they tend to become sweet at lower temperatures.

For the prolonged storage of green vegetables the conditions required, according to W. H. Smith,[2] are more exacting than for top fruits since vegetables become very unattractive when wilted. The store must be particularly well insulated and the cooler must have "sufficient surface area . . . to absorb both the heat produced by the produce itself and the heat that passes through walls and ceiling without employing an excessively low temperature in the refrigerant. . . . In some cases it may prove advisable to provide a pre-cooling room large enough to accommodate the maximum daily input." Careful attention should be paid to air-circulation and the arrangement of the material, and the R.H. should be high—90 to 95 per cent. Even the containers should be sufficiently saturated with moisture to prevent them from absorbing water from the produce; on the other hand they should not be soaking wet. Smith emphasizes that stored vegetables will have lost some of their "staying power" and will not keep out of store as long as freshly harvested produce. In ordinary cool storage cauliflowers will keep about 3 weeks at 32° F. to 34° F. and 90 to 95 per cent R.H. during which time they lose about 3 per cent of their weight. Storage above this temperature is inadvisable except for much shorter periods.

Meat

Cool storage or "chilling" of meat is not complicated by the fact that the tissues are still alive. Although there are still undestroyed enzymes in chilled meat which can cause autolytic changes, the main cause of tainting is the surface growth of micro-organisms. It is possible, however, to preserve meat in good condition at 29½° F. for about 30 days *if proper hygienic precautions have been taken to minimise surface contamination during slaughtering and handling.* This is long enough for transport from Argentina (about 3 weeks) with a margin to spare for distribution to consumers in this country.

Poultry

In the United States a very large trade in chickens about 10 to 12 weeks old and weighing about 3 pounds ("broilers") has

been made possible by cooling them down to 34° F. before distributing them in refrigerated cars or in ice. Those who have experience of this trade consider that the lessening of wastage by chilling far outweighs any disadvantages caused by loss of "bloom" through condensation. But, as Brooks[3] has pointed out, a fresh bird has only a short storage life; if they are to be kept very long they must be frozen (see p. 149). The onset of tainting is said to be detectable in these young birds in 24 hours at ordinary temperatures. The first visible sign is the appearance of a bile stain on the liver, but other organs soon become involved and the vents often become green.

Eggs

Shell eggs are stored at 30° F. to 32° F. and at a R.H. controlled at 85 per cent to check mould growth on the shells. Losses from bacterial rotting begin to appear after 3 months and "off" flavours in otherwise sound eggs at 5 months. There is also an enlargement of the air-space, a gradual thinning of the whites and a weakening of the yolk membrane. Washing eggs greatly increases the loss due to bacterial rotting.[4] Eggs which have been stored should be brought up to 50° F. in moving air and consumed promptly.

Dairy Products

These are mostly carried at ordinary temperatures for short voyages. Cheese becomes fragmented by freezing and can be carried at 45° F. on long voyages. This temperature permits ripening to continue slowly. Butter from New Zealand is frozen at 10° F. to 15° F.

REFRIGERATED GAS STORAGE

It was discovered in experiments on the cool storage of apples that if carbon dioxide was allowed to accumulate to excess in the store, through respiration, some varieties developed a disease known as "brown-heart." After many cargoes had been lost in this way it was found that for many varieties of apples there was an intermediate stage between a normal and an exhausted atmosphere in which they could be reduced almost to a state of suspended animation, as regards respiration and general

metabolism, and have their storage life correspondingly length-
ened without damage.

Fruits

So far as fruits are concerned, gas storage has only been applied
commercially to apples and pears, but recent experiments with
bananas show promise, provided that a method can be found for
keeping the store clear of ethylene.[5]   In the case of Bramley
Seedling apples an atmosphere of the correct composition is
built up in a sealed store at 40° F. by the respiration of the apples
themselves and the advantage gained by ordinary cool storage at
38° F. to 40° F. is usually more than doubled.   Other apples
require other gas mixtures and the conditions necessary for
several important varieties have been described by Kidd and
West.[6]   Thus, for Cox's Orange Pippin and Ellison's Orange
the best gas mixture consists of $2\frac{1}{2}$ per cent O, 5 per cent CO_2 and
about 92 per cent N. and this can only be maintained by means of
absorbers containing milk of lime or caustic soda.   Without this
adjustment brown-heart would develop.   Newton Wonder,
Blenheim Orange and a few other varieties require good ventila-
tion and cannot be gas stored.

For pears, as for apples, the gas mixtures and storage tem-
peratures again depend on variety, and the conditions for pears
have also been described by Kidd and West.[7]

Vegetables

Smith[8] has shown that storage in an atmosphere containing
10 per cent CO_2, 11 per cent O, and 79 per cent N. at 32° F. to
34° F. preserves cauliflower and broccoli about 2 weeks longer
than ordinary cool storage at 32° F.   Gas storage does not suit
carrots, which require good ventilation.

Meat

The spoilage of meat through the formation of bacterial slimes
and growths of moulds and yeasts on the exposed surfaces
precedes spoilage through oxidative and enzymic changes in the
fats, and a concentration of 10 to 12 per cent of CO_2 in the
atmosphere of a ship's hold has been found sufficient to inhibit
the growth of micro-organisms long enough (6 or 8 weeks) to
permit meat to be carried at $29\frac{1}{2}°$ F. from the Antipodes in

satisfactory condition. Higher concentrations of CO_2 give still longer protection but the colour of the meat is affected through the formation of methaemoglobin.

Eggs[9]

Sixty per cent of CO_2 in the atmosphere of an egg store containing unwashed eggs prevents the growth of micro-organisms for 9 months and there is no need to control the humidity. Unfortunately, however, the whites gradually become thin and watery, which is a drawback if the eggs are fried or poached. With low concentrations of CO_2 (2 to $2\frac{1}{2}$ per cent) the whites remain thick but the same humidity control is necessary as with eggs stored in air, if surface growth is to be prevented. Eggs that have been gas-stored should be allowed to stand about 2 days to allow the dissolved CO_2 to diffuse out. This also gives an opportunity for the shells to dry thoroughly.

All gas stores should be practically gas-tight and are often lined with metal sheets. When ventilation is necessary it must be rigidly controlled and instruments must be installed to indicate whether the correct type of atmosphere for the product stored is being maintained.

FROZEN STORAGE

(1). FRUITS AND VEGETABLES[10]

In frozen storage the life processes of fruits and vegetables no longer constitute a problem: the act of freezing stops them entirely and they are never resumed. Micro-organisms also become inactive for all practical purposes at from 23° F. to 20° F., and although their spores are not killed even at the lowest freezing and storage temperatures they do not become active again until the material is thawed and allowed to stand. The problems which remain and come to the fore are concerned with rates of freezing, surface drying, thawing conditions, the effect of freezing on the material and with temperatures of storage in relation to the activity of undestroyed enzymic systems and to direct oxidation.

Plant tissues vary considerably in the degree to which they can be sub-cooled without actually freezing. Juicy, sappy tissues are the most susceptible and dry, woody parts and seeds the least;

hence most of our edible fruits and vegetables freeze fairly readily. The full damage due to freezing does not become apparent until plant tissues have thawed, when juicy fruits with thin skins, like strawberries, lose their attractive appearance and collapse, with loss of juice, under their own weight. Thicker skinned fruits, like gooseberries and cherries, and also green vegetables, become soft and flabby.

These results are inevitable, being due to the disruption and denaturation of the colloidal constituents of the cells which render them incapable of retaining juice.

Rate of Freezing

Very rapid freezing, with its effect on the size of ice crystals, can lessen the degree of collapse of plant tissues and the amount of liquid exuding from them and this may be a permanent advantage with a few products, e.g., asparagus, but, usually, the effect, such as it is, is ephemeral, and becomes negligible with most fruits if they are allowed to stand long after thawing. Most authorities find that if fruits and vegetables are frozen in anything from 1 to 7 hours the results are satisfactory. The condition of the material before freezing is a much more important factor.

Temperatures for Storage

As indicated above, the primary consideration in deciding the temperature at which to store frozen foods is whether it will have a sufficient retarding influence on chemical activity to preserve them in attractive and acceptable condition for the anticipated duration of storage. For general purposes, it is considered that, except, possibly, for short periods of 1 or 2 months, when 10° F. to 14° F. may be low enough, the storage temperature for fruits and vegetables should not exceed 0° F. or, preferably —4° F. or — 5° F. At these temperatures storage from one season to the next is quite possible.

The forms which deterioration can take depend greatly on the material in question. The act of freezing causes, so far as is known, no chemical change. At the same time, it does not destroy the enzymes and other unstable substances which exist in living material. These are curtailed in their activity partly by the low temperature and partly by the immobilisation of the water of the cell sap when it freezes. They do, however, react

slowly and at 14° F. we find, for example, after a few months, that pectic enzymes have reduced the setting power of fruit for jam-making perceptibly more than at 0° F. Also the pigments of red fruits will have darkened more, the skins of gooseberries and blackcurrants will have become tougher and there will be more general discoloration of fruits which turn brown when bruised.

Procedure for Fruits

Since about 1907 carefully sorted and graded soft fruits (strawberries, raspberries, &c.) have been intimately mixed with dry sugar (1 or 2 parts of sugar to 4 or 5 parts of fruit) and frozen in barrels or other large containers for catering or making into jam. In the best practice the barrels were frozen in a "sharp freezer" at —10° F. to —20° F. and transferred to a permanent store at 0° F. or 10° F. Later, considerable quantities began to be packed in smaller containers for institutional or household use and this has grown into the modern "frozen pack" industry in which cartons, lined inside and covered outside with impermeable film, are frozen rapidly by mass-production methods and delivered to consumers in the frozen state. The sugar or sugar syrup with which the fruit is covered, to some extent inhibits enzymic and oxidative changes. It also disguises the collapse of the tissues and penetrates them, thus rendering the product more presentable and acceptable for eating, thawed or half-thawed, with cream or ice-cream.

Soft fruits, which do not turn brown on thawing, are by far the most suitable for frozen pack methods. Apples and pears which soften and turn brown rapidly are quite unsuitable. Stone fruits are frozen commercially to some extent but should be consumed before they turn brown, although it is claimed in some quarters that browning of sliced peaches can be prevented by adding ascorbic acid to the syrup in which they are frozen.

Procedure for Vegetables

It is not feasible to freeze raw vegetables because, except for a few, they contain enzymes that produce unpleasant flavours, colour changes and toughening during storage and thawing, no matter what the storage temperatures may be. These enzymes can be destroyed and the vegetables rendered relatively stable, both chemically and physically, by scalding or "blanching" them

in boiling water or steam for periods depending on the size of
the pieces; thus, peas take $2\frac{1}{2}$ to 3 minutes, sliced runner beans 2
mins. and larger pieces, like cut-up broccoli, 3 to 4 mins.   The
scalding is usually a continuous operation and is followed immedi-
ately by rapid cooling in cold water, packing into flat, rectangular,
moisture-proof cartons, similar to those used for fruit, freezing
rapidly either in an airblast or in a plate freezer and storing at
0° F. or −5° F.   Delays and stoppages between all these proces-
ses are even more important to avoid than with fruits.

Some loss of soluble nutrients is unavoidable during scalding,
but this amounts to little more than in household practice and to
no more than in canning, particularly if as little scalding and
cooling water as possible is used.

There is no need to allow the vegetables to thaw out for
consumption; they should be heated up directly from the frozen
state and, when cooked, they should be practically indistinguish-
able from cooked fresh vegetables.

2.   ANIMAL PRODUCTS

Although life processes have ceased in the flesh of animals
which have been slaughtered, raw meat contains undestroyed
enzymes, some of which are beneficial and can render the meat
tender if it is hung for a time before cooking, while others can
turn the fat rancid.   Direct oxidation can also turn fat rancid
and the outside of the meat brown.   Unlike fruits and vegetables
which are protected from "freezer-burn" by packaging, meat
has, in the past, always been exposed and has therefore suffered
in appearance on prolonged storage.   It is obviously expensive
and difficult to provide impermeable wrappings for sides and
large joints and the problem could be eased considerably if all
meat were deboned before shipping and pressed into compact
forms according to the most modern practice.   This would also
save much shipping space.

Meat

As with fruits and vegetables, enzymic and oxidative changes
can proceed in frozen meat at rates depending on the storage
temperatures but these are slow enough for most practical pur-
poses at 14° F. which has long been recognised as the "com-
mercial" storage temperature for meat.   Recently, however, it

has been suggested that, with improved refrigeration equipment, lower temperatures (−10° F. or 0° F.) might be adopted with advantage.

The hygienic handling of meat, both before and after freezing, is of the greatest importance. Micro-organisms are not killed by freezing; and some further contamination is unavoidable in distribution. Moreover the surface of meat which has been frozen and thawed is an excellent medium for bacterial growth. This is partly because of unavoidable condensation but, more especially, because, as in the case of plant tissues, the colloidal structure of meat is changed so that it can no longer retain all its fluid content. There is therefore exudation of fluid or "drip" which provides a damp, nutrient surface and is very pronounced in whalemeat, less so in beef, and much less so in mutton and pork.

Fish

With the principal fishing grounds now far distant from the nearest ports, the question of freezing fish at sea is becoming of increasing importance and it seems probable either that trawlers will be equipped for suitably gutting, filleting *and freezing* fish immediately after catching or that large factory-ships equipped for this purpose will accompany the fishing fleets. The chief difficulty in carrying out such a project is the great freezing capacity needed, not only to freeze large catches but to freeze them quickly, as it has been shown that if fish are not frozen solid within about 2 hours they become soft and wet on thawing and of poor texture when cooked. Fish also require a lower storage temperature than meat, i.e., not above 0° F. or −5° F.

Poultry

According to Brooks,[11] "sharp freezing" at −10° F. to −25° F. and storage at 0° F. or slightly lower, satisfies all the normal commercial requirements for poultry. Even if they are to be stored for short periods the storage temperature should not be higher than 10° F. All birds should be bled but whether they should be eviscerated or not is a matter of convenience: it makes no difference to quality. To prevent freezer-burn, it is necessary to use an efficient wrapping material.

Eggs are never frozen in the shell but much egg pulp is frozen in tins and imported for catering. The pulp should be frozen promptly and stored at 0° F. or slightly lower.

Cooked and Prepared Foods

In a sense, scalded vegetables can be regarded as partly cooked foods, and following from this, a great many fully cooked foods are now being frozen and stored. The chief difficulty is with fatty foods which tend to turn rancid, even at low temperatures; hence protective wrappings and careful packing are necessary. Wrappings can also prevent freezer-burn.

T. N. M.

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CHAPTER 12

DEHYDRATION

THE protection of foodstuffs from spoilage by moulds and bacteria is a major concern of the food technologist. One of the most obvious, and most ancient, methods of accomplishing this is to dry the food to such an extent that mould or bacterial growth cannot occur. The accompanying reduction in weight and volume make foods preserved in this way especially attractive for military purposes, and much of the development of modern dehydrated foods has been a result of the stimulus provided by the Second World War.

The term dehydration is applied to artificially induced drying, as distinct from drying in the sun or wind. A very wide range of food products are dehydrated today; among them are apricots, peaches, apples and other fruits (mainly in the United States), potatoes, cabbage, carrot, beetroot, parsnip and other vegetables; malt, milk, eggs, meat, fish. There are also numerous special products such as baby foods based on dehydrated milk, and soup powders.

It will be impossible in this chapter to describe the technology of so wide a range of products, and further information about many must be sought in the literature mentioned in the bibliography. The outlines that follow are confined to a very few commodities, but have been selected to illustrate typical dehydration processes and some of the underlying principles. Recent developments are described in the final section.

DEHYDRATION OF VEGETABLES AND FRUIT

(a) Vegetables[1]

Raw vegetables of high initial quality are trimmed and cut into portions of a size suitable for drying: the dimensions of the portion are very important as they affect the rate and extent of drying and the rate of reconstitution. Root vegetables are commonly cut into strips of cross-section area $\frac{3}{16}$ in. $\times$ $\frac{3}{16}$ in., or
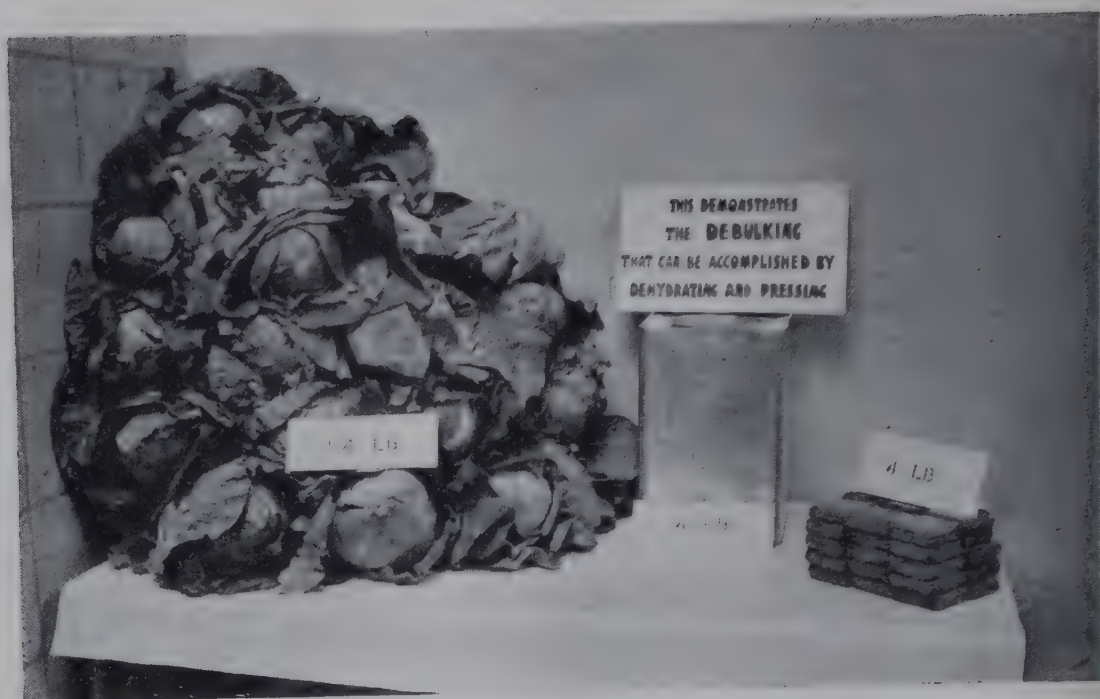
FIG. 1.—DEHYDRATION OF VEGETABLES.*

Dehydration is not only a method of preservation of food, but brings about great reductions in bulk and weight, which are particularly important for military purposes. One cwt. of cabbage, for example, is reduced to about 4 lb. by dehydration and can be packed in a 4-gallon can. Compression reduces the bulk still further.

dice, generally not exceeding $\frac{1}{4}$ in. or at the most $\frac{3}{8}$ in., while leafy vegetables are shredded.

Blanching or Scalding

The cut vegetable is passed through a blancher or scalder in which it is immersed in near-boiling water (or scalded in steam) for 2 to 3 minutes: this inactivates enzymes and effects a measure of pre-cooking which improves the texture of the finished product. The addition of sulphite at this stage improves the storage qualities of most vegetables. When green vegetables are scalded, it is necessary to neutralise the organic acids liberated from the plant cells during scalding (and even to keep the liquor slightly alkaline) since in hot and acid conditions chlorophyll becomes chemically altered and assumes a dull olive colour.

Leaching of solutes from plant tissues can be very severe during water scalding, but if the leached solutes are allowed to accumulate to some extent in the scald liquor wastage is reduced. Too high a concentration of solutes in the scald liquor, however, may lead to discoloration of the product and difficulties during drying

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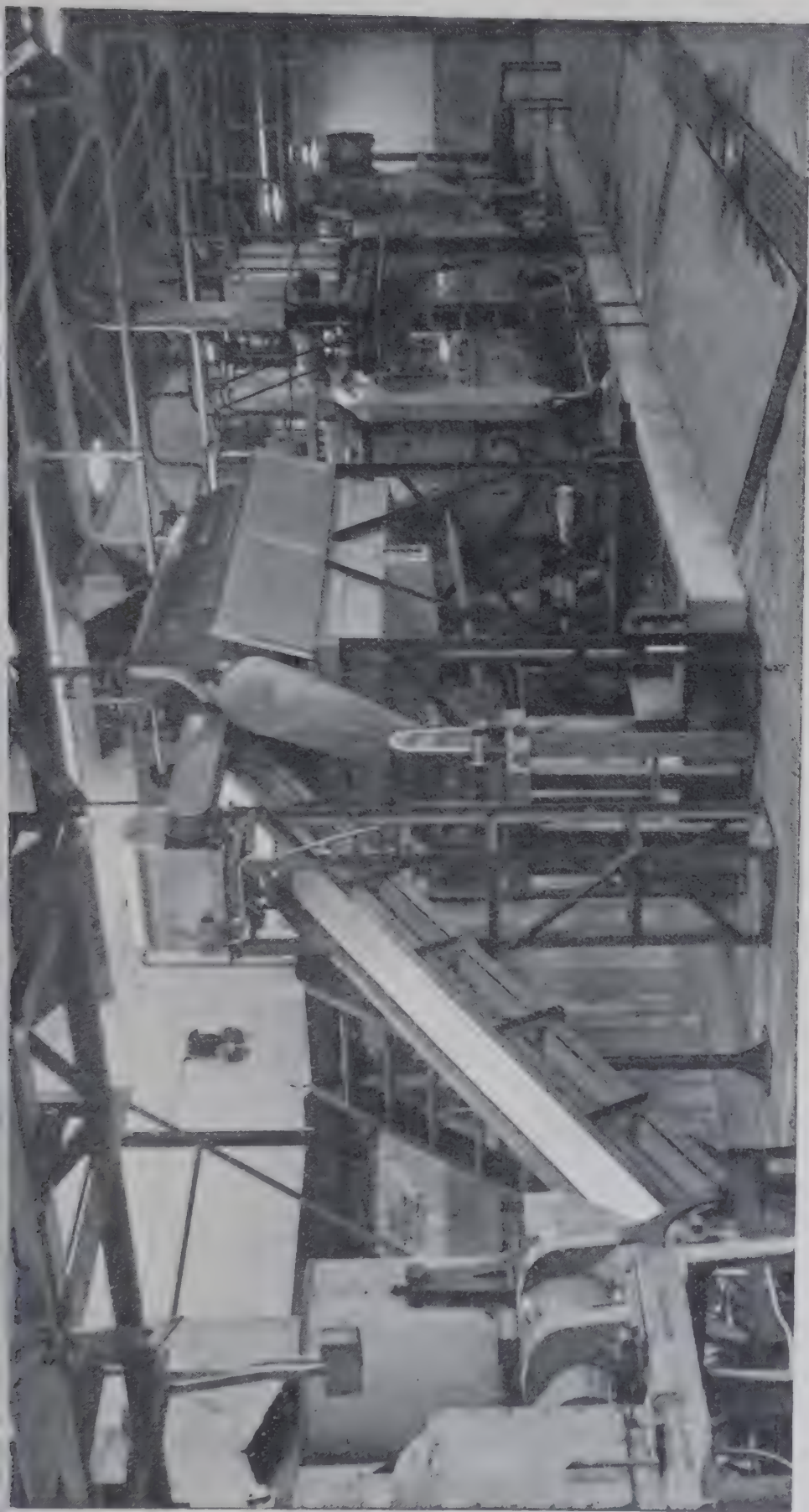
FIG. 2.—VEGETABLE DEHYDRATION.

Before dehydration vegetables are trimmed, cut into shreds, strips or dice, washed, scalded (blanched) and cooled. The illustration shows, from left to right, a strip or dice cutter; a cabbage shredder; conveyor; stainless steel rotary washer; blancher (centre shaft, stainless steel drum) with tanks above for holding dosing solutions of sulphite or carbonate; and cooler.
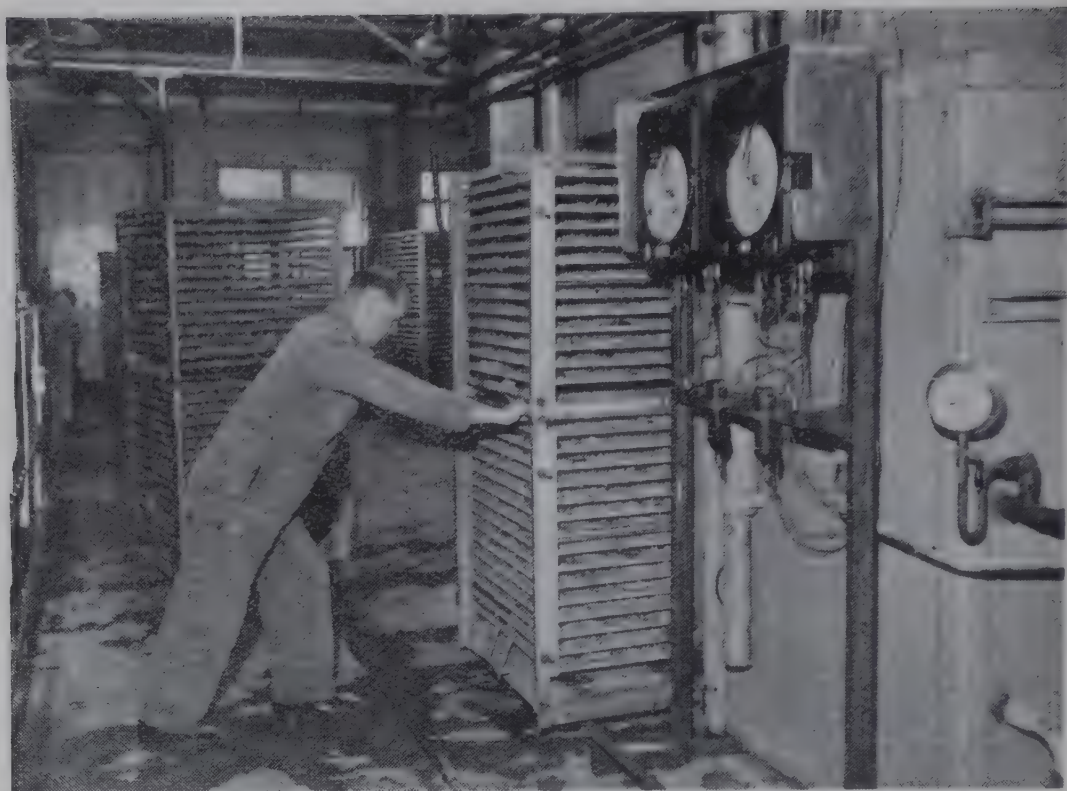
FIG. 3.—VEGETABLE DEHYDRATION.

Blanched vegetables, on wire mesh trays stacked in trolleys, are pushed
into a drying cabinet: the heated air is blown across the trays.

and storage. Scalding in steam instead of hot water reduces the
extent of leaching but may have adverse effects on the storage life
of the product.

Drying Process

The actual dehydration is usually carried out in a circuit of
heated air which may be blown over trays containing the vegetable
in cabinets or tunnels, or through the vegetable carried on a
perforated conveyor belt. Drying is frequently completed in
deep trays or in "bins" through which the heated air is blown.
In the early stages of drying, if the wet bulb temperature is
sufficiently low, the dry bulb temperature of the air can be high
(over 210° F.); the rapid evaporation of moisture keeps the
temperature of the vegetable at that of the wet bulb and heat
damage does not ensue. After a short initial period, however,
the rate of drying begins to slow down and becomes governed
by the rate at which water can diffuse from the centre of the
portion to the outer surface. Consequently the temperature of
the material rises above that of the wet bulb, and approaches the
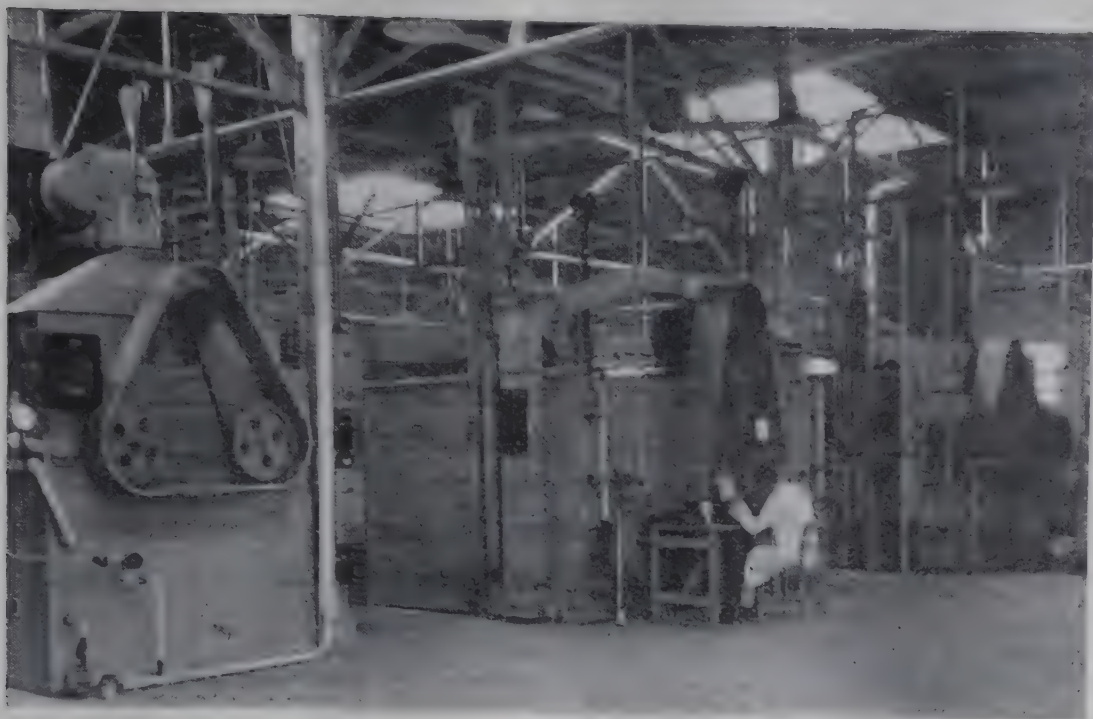
FIG. 4.—VEGETABLE DEHYDRATION.—A THREE STAGE CROSS-FLOW DRIER.

The first cabinet holds one batch (two trolleys carrying some 500 lb. blanched vegetable), the second and third four batches each. A typical drying cycle (e.g. for cabbage) is for a batch to have $\frac{1}{2}$-hour in the first cabinet with dry bulb temp. 210° F. and wet bulb temp. 135° F.; 2 hours in the second cabinet with dry bulb temp. 165° F. and wet bulb temp. 125° F. and 2 hours in the third cabinet with dry bulb temp. 145° F. and wet bulb 90° F.

dry bulb temperature, and it is necessary gradually to reduce the dry bulb temperature of the surrounding air from the initial high level to a final maximum of 140° F. to 150° F., though prolonged drying at lower temperatures may be necessary if exceptionally low moisture contents are desired.

For powders, purées, etc., other forms of drying are employed—pneumatic driers, spray driers and roller driers all find application here.

(b) Fruit[2]

In the United States fruit dehydration is a major industry. Until comparatively recently sun-drying was generally used, but present-day practice is to use some form of hot-air drier. In some cases "natural draught" air circulation is used, with the heated air rising by convection through banks of trays carrying the fruit; in others the heated air is circulated by fans in cabinets, tunnels or conveyor driers similar to those described for vegetable

dehydration. Some fruit, for example, peaches and apricots, are sulphited, often by exposure to the fumes of burning sulphur. There still appears to be some doubt about the necessity of scalding, as some fruit are not scalded, while other fruit may or may not be scalded, according to the views of the particular manufacturer. In Britain[3] fruit dehydration has been mainly experimental, and moderately successful attempts have been made to use hop-kilns for this purpose.

It is generally not practicable to cut fruit into portions as small as those used for vegetables, and consequently drying may be prolonged. Half peaches, for example take 15 to 24 hours to dry and even apple rings take 6 to 10 hours. Current research (*see* p. 159) is attempting to reduce these figures.

DEHYDRATION OF ANIMAL PRODUCTS

(a) Fish

Raw fish cannot be successfully dried in a current of hot air. The drying time is long, the product does not properly reconstitute, and it develops cured flavours. A greatly improved product can be obtained by *pre-cooking*. Cooking removes some of the water from the fish and followed by mincing permits the exposure of thinner and more uniform layers, thus leading to more rapid drying.

Briefly, the standard process is to mince the fish after pressure cooking, and spread on to wire mesh trays. The juice is discarded as it would give a soggy mince if added back to the cooked fish. A light granular material with a large surface area to aid evaporation is desirable. Hot air is circulated over the mince in a cabinet, and drying occupies about 4 hours. Dry bulb temperature is initially about 85° C. and wet bulb 55° C., the temperature falling during the drying period to a final 65° C. to 70° C. Initially the fish attains the wet bulb temperature and during the process gradually approaches the dry bulb temperature.

Gadoids and flat fish yield acceptable products, but herring develops a rather oily flavour which can be masked by using smoked fish, e.g. kipper. The presence of up to 15 per cent fat in the raw fish does not appreciably increase the drying time.

Round cod will yield about 40 to 45 per cent of its weight as

fillets, and the weight of dehydrated cooked mince obtained is about 16 per cent of that of the fillets.

Feeding tests on rats have shown that dehydration does not affect the nutritive value or digestibility of the fish.

(b) Meat[4]

As is the case with fish, hot air drying of raw meat does not give a good product. It becomes case hardened and forms a solid mat thus decreasing the surface area exposed to the stream of hot air. This results in a long drying time and inferior quality.

The rate of drying of any foodstuff in hot air is markedly affected by particle size. Small portions dry most rapidly because of a high surface area to volume ratio and also water has only a short path from the centre to reach an evaporative surface. Thus it was found that a cooked mince yielded the best product; case hardening was avoided and drying was completed in about 4 hours. Brief details of the process developed in this country, and used in several meat exporting countries during the Second World War are given on the following page.

FIG. 5.—MEAT DEHYDRATION.
For drying in a current of hot air, the mince is spread on wire mesh trays.
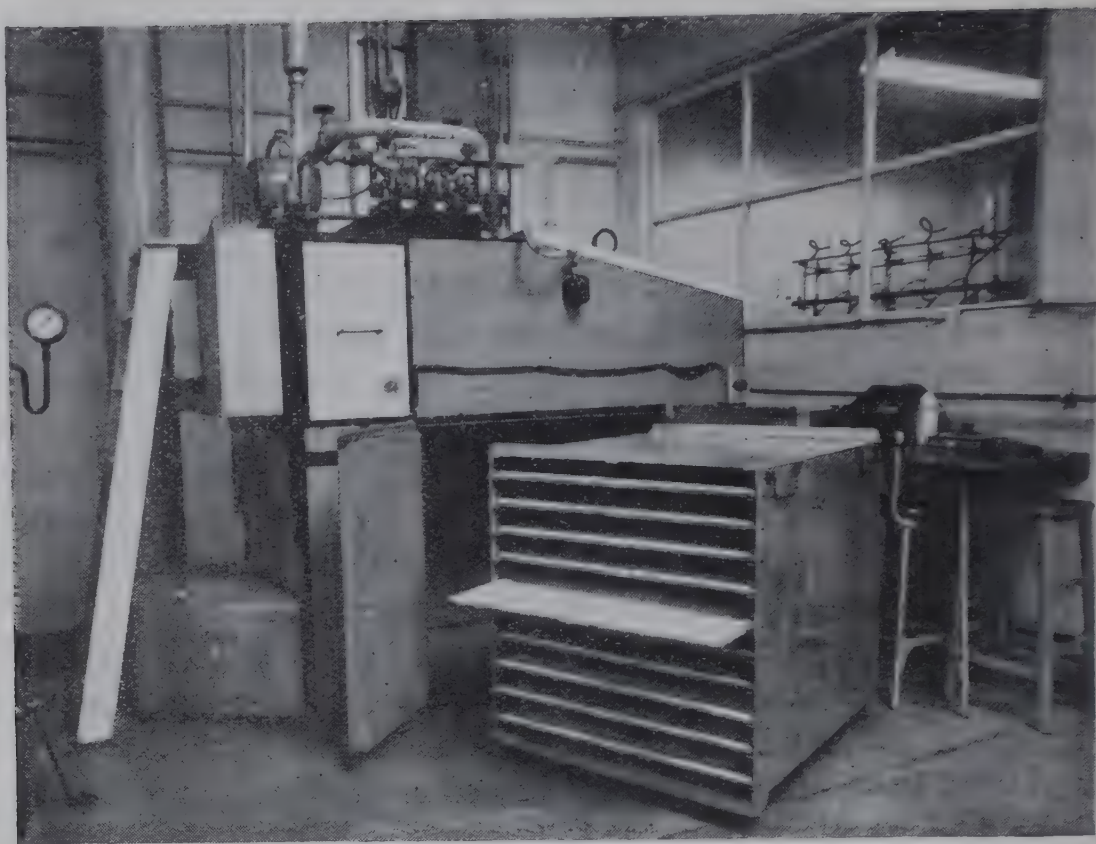
FIG. 6.—MEAT DEHYDRATION.

This overdraught dryer is used mainly for meat dehydration, but is also applicable to other commodities.   The trolley holds 20 trays, each 18 in. × 32 in. and when filled accommodates about 150 lb. minced cooked meat.

The meat is boned out, trimmed free of excess fatty tissue and cut into approximately 2 in. cubes.   The cubed meat is cooked in boiling water in an open pan until just brown throughout, then is removed and cooled by a stream of filtered air in a bin cooler. The remaining gravy contains an appreciable proportion of the flavouring substances of meat and must be included in the product if it is to retain its full flavour.   The fat is separated from the gravy and may be set aside for adding to the dried product if a high fat content is desired.   The aqueous phase is concentrated to about one quarter of its original bulk and poured over the cold meat chunks immediately before mincing.   The mince is spread as a loose and open layer on wire mesh trays at about 2 lb./sq. ft. and loaded into an over-draught cabinet drier.

The dry bulb temperature is set at 176° F. for the first hour, and thereafter held at 160° F.   The dampers are adjusted to give a wet bulb reading of 125° F. over the first half hour.

Suitable meat for the process is carcases of lean steers and heifers, dressing out at 55 to 50 per cent, giving a yield of standard dehydrated meat (40 per cent fat and $7\frac{1}{2}$ per cent water content) equal to 23·5 to 25·8 per cent of dressed carcase weight. Drying is influenced by the degree of fatness of the meat, and trimming should aim at giving a dry product containing 30 per cent fat.

Metabolic experiments[5] on rats have shown no detrimental effect on the biological value of the proteins.

RESEARCH AND RECENT DEVELOPMENTS

Apart from the use made of dehydrated products by the armed Services, developments have been made in the civilian sphere. Dehydrated vegetables and soups based on dehydrated materials are on the market and slowly gaining in popularity. Quick reconstituting mixes have recently been demonstrated.

Food dehydration is the subject of a considerable amount of research: the fundamental aspects of scalding, drying and storage are being studied, and investigations are being pursued into alternative manufacturing techniques, particularly for scalding and drying. The three main forms of deterioration of dehydrated foods, viz. (1) Rancidity and other oxidative changes, (2) Non-enzymic browning, and (3) toughening and texture changes, are being examined and efforts are being made to obtain longer storage life.

The most suitable pack for dehydrated foods is the can. Alternative methods of packaging are being investigated, particularly those using plastic films and laminates. Their usefulness is governed generally by their resistance to perforation by hard edges or corners of dehydrated foods, and their oxygen and water vapour transmission characteristics.

Foodstuffs dehydrated as small portions are nutritious and palatable products, but their usefulness is limited. Efforts are being directed therefore to the dehydration of larger and more usual portions of foodstuffs (e.g., steaks, chops, fish fillets, slices of root vegetables and whole fruits). A vacuum drying technique is necessary if a prolonged drying time and marked deterioration in texture and flavour are to be avoided. Drying at low temperatures is also permitted by such a process with accompanying

improvements in quality of the product. The three methods of vacuum drying under most active investigation are:

1. The Vac-ice process (freeze drying).
2. Vacuum contact-plate process.
3. New Zealand process (at the moment only applied to meat).

None of the processes has yet been adopted commercially but commercial sized pilot equipment of (2) is under active investigation, and a small pilot plant examination of (3) has been completed.

1. Vac-ice Process

This is the most expensive process and is being most actively studied in the United States.[6] It is doubtful at the moment whether it can be economically applied to foods.

The foodstuff is frozen and the water now present as ice is removed by sublimation in a high vacuum. There are two stages in the process—(a) the ice is evaporated from the frozen mass, (b) after warming to about 35° C. to 40° C. the remaining moisture is removed. During the first step some 95 per cent of the water is lost and in the second the water content is reduced to 0.5 per cent.

The plant consists of a vacuum shelf drier and a means for providing the vacuum and removing water vapour. There are three general methods for removal of water vapour—(i) condensation using dry ice or refrigerants. Non-condensable gases arising from air dissolved in the tissue and leaks etc. must be removed with a pump. (ii) Regenerable desiccants, e.g., silica gel. Heat is produced during water adsorption, and the desiccant must be cooled by special means. Specially prepared calcium sulphate in controlled amounts may be employed without cooling. (iii) Direct pumping. The volume occupied by the water vapour under expanded conditions in high vacuum is enormous, so the pump must have a high volumetric capacity. In large scale operation multi-stage steam ejectors are preferred.

During the first stage of drying where an efficient means for removal of water vapour is available, the controlling factor for rapid evaporation is the speed at which heat can be carried to the icy surface through the poorly conducting foodstuff.

Generally speaking, with products of average characteristics drying is at the rate of 1 mm. depth per hour at –18° C. and 250
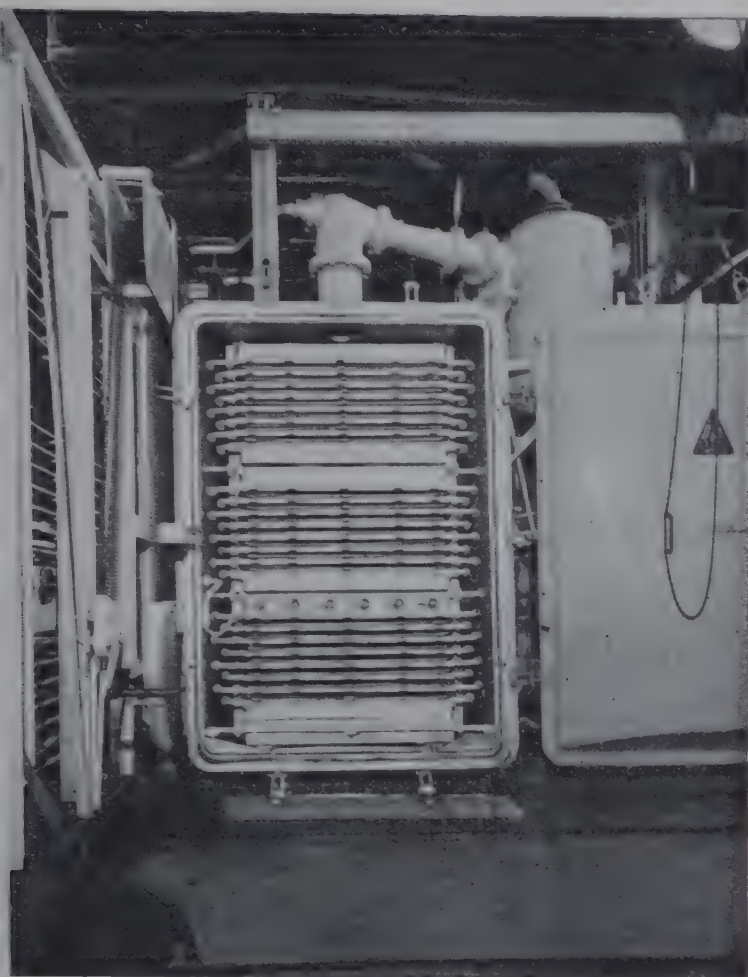
FIG. 7.—A COMMERCIAL SIZED CONTACT PLATE DEHYDRATION CABINET.

The foodstuff is placed on an aluminium tray (Fig. 8), covered with a sheet aluminium lid and inserted between the heating plates. The ends of the trays and lids can be seen in the illustration. The heating plates are in three banks of eight, allowing the insertion of 21 trays; the plates in each bank can be moved together, concertina fashion, to maintain thermal contact as drying proceeds. A cabinet of this size holds $\frac{1}{2}$ to 1 ton of foodstuff, according to the tray loading density adopted for each commodity.

microns pressure. Under these conditions the rate of drying is constant during the first stage, i.e., until all the ice is gone. This requires about 80 per cent of the total drying time and removes about 95 per cent of the total weight of water. During the remaining 20 per cent of the time the final moisture content of 0·5 per cent or less is achieved.

The product is a very porous solid, identical in volume with the initial material. The structure confers almost immediate reconstitution in cold water, in many cases requiring only 5 minutes contact. The very low temperatures employed minimise damage to labile substances and loss of volatile constituents; also bacterial growth and enzymic action are inhibited. The reconstituted product approximates closely to the texture and flavour of the starting material.

2. Vacuum Contact Plate Process[7]

The vacuum for this process is obtained by steam ejectors. The steam is condensed in a jet condenser from which the
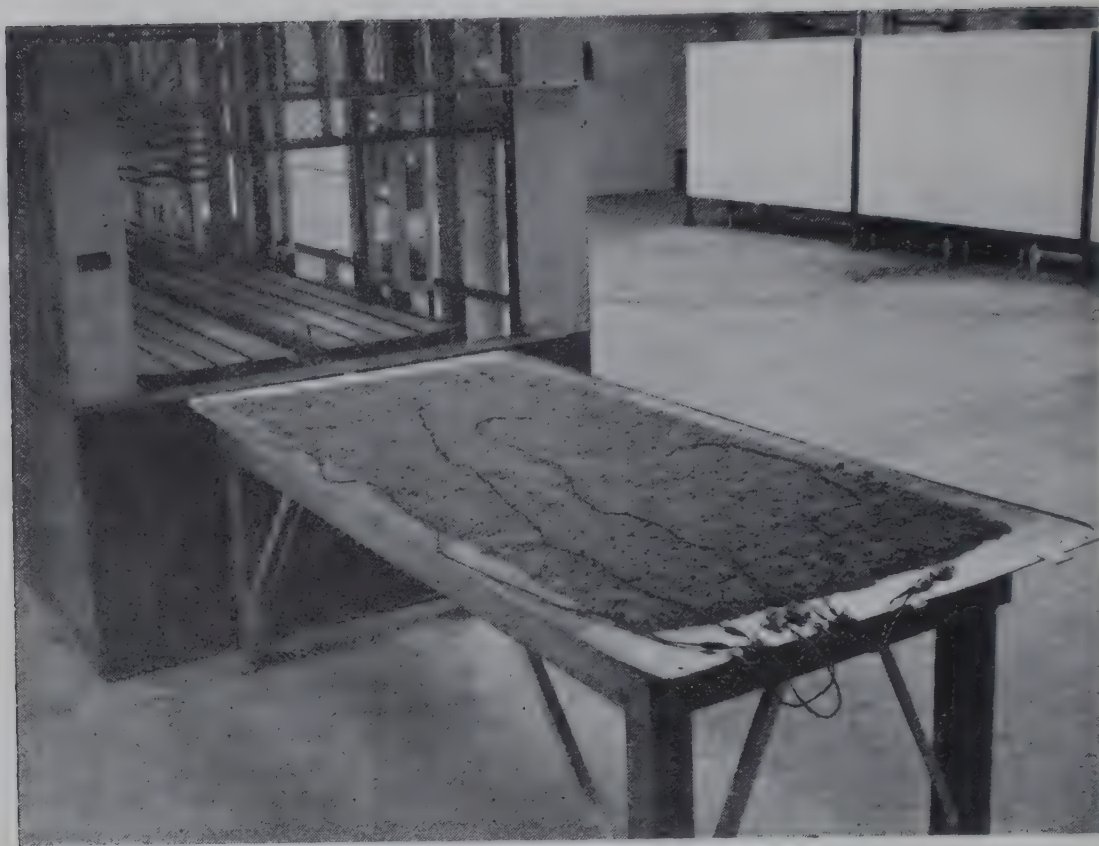
FIG. 8.—MINCED MEAT SPREAD ON A TRAY BEFORE DEHYDRATION IN THE
LARGE VACUUM CONTACT-PLATE CABINET SHOWN IN FIG. 7.

Thermocouples for measuring the temperatures actually attained by the meat
during processing are in position.

non-condensable gases are removed by twin water ejectors; the
cooling water and condensate are removed by a centrifugal pump.
At the beginning of the process when evaporation is most rapid
the pressure in the dehydration cabinet is about 10 mm. of
mercury, and in the final stages the pressure falls to $2\frac{1}{2}$ mm.
mercury. The cabinet contains a series of hollow steel plates
with internal labyrinths through which hot water is circulated.
The plates can be closed together to sandwich the foodstuff
between them, thus achieving a good heat transfer into the
material and leading to rapid evaporation. As the food shrinks
due to water losses continuous contact can be maintained by
further closing of the plates.

The foodstuff is laid on sheet aluminium trays (at 2 to 4 lb. per
sq. ft.) to form a layer of uniform thickness, covered with a
similar lid and fed between the heating plates. The cabinet is
evacuated and heating water circulated through the plates. The
temperature of the water is controlled to maintain the desired

temperature in the foodstuff. In practice the latter is considerably lower for most of the drying period than is normal in hot air drying methods. Moisture levels of 4 to 6 per cent are reached in 4 to 8 hours depending on the material and density of loading.

The products when reconstituted and cooked are attractive, the flavour and texture of many being indistinguishable from the normal cooked food.

3. New Zealand Process

This process is similar in many respects to the Zimmermann process[8] developed by an Austrian of that name during the Second World War and also by Platt and Heard in the United Kingdom.[9] It has been employed in New Zealand solely for the dehydration of meat. Trimmed boneless meat is fed into molten fat contained in the dehydrator, which is a horizontally rotating jacketed steel vessel heated by water at about 200° F. The molten fat conducts the heat to the meat and drying is effected at a pressure of 1 in. of mercury. Drying to about 3 per cent of water takes 4 hours. At the end of this time the meat is removed from the dehydrator and the molten fat which permeates the meat is removed in a hydro-extractor. Reconstituted in water, a palatable product is obtained for pot roasting or cooking in a casserole.

E. G. B. G.
E. J. R.

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PART 2.—LABORATORY CONTROL

CHAPTER 13

SUGAR REFINING

THE items of analysis for raw sugars customarily reported are the following:

1. Polarisation (Pol.)
2. Reducing Sugars (I.)
3. Moisture
4. Ash
5. Sucrose
6. Organic non-sugar

1. Polarisation

This is a commercial test for the approximate content of sucrose in raw sugars and is the figure on which buying and selling and customs duty is assessed. The saccharimeter used for measuring the polarisation is a polarimeter graduated on the International Sugar Scale in °Sugar or °S. The normal weight 26·0 gm. of purest Sucrose (dry and corrected for the traces of ash, reducing sugars and organic non-sugar) when dissolved in water and made up to the mark at 20° C. in a 100 ml. polarisation flask will give a reading of 100°S. with a 200 mm. tube in the saccharimeter.

To polarise a raw sugar (cane or beet) 26 gm. of the sample are weighed out in a flat bottom nickel dish provided with a spout and partly dissolved with cold distilled water using a rounded glass rod as stirrer. The partly dissolved sugar is poured into a 100 ml. pol. flask and a jet of distilled water from a wash bottle used to rinse the dish and rod into the pol. flask. Rotary shaking completes the solution of the sugar and further water is added with rotary mixing until the volume is approximately 70 ml. A predetermined quantity of basic lead acetate solution is added drop by drop with vigorous shaking to precipitate non-sugars, coagulate colloids and remove some colour and is followed by some alumina cream. Flask and contents are brought to 20° C. in a bath and final adjustment to mark is made with water at 20° C. Final mixing is achieved by inverting the flask several times with the top covered with the thumb.

The solution is then filtered through a pleated filter paper, held in a glass funnel with cut off stem and resting in a glass beaker or stouter

glass lipped receiver and covered with a clock glass. The filtered solution should be free from any trace of turbidity or difficulty will be experienced in the subsequent operation. The 200 mm. pol. tube previously cleaned is rinsed with a little of the solution and then filled, taking care to eliminate any adherent air bubbles. The glass cap is replaced and the top screwed on, being held tight by the rubber washers which at both ends prevent strain of the glass cap which would alter its optical properties. The optical rotation of the solution is then taken in the saccharimeter and the result corrected for zero reading, is the polarisation of the raw sugar.

QUANTITIES OF BASIC LEAD ACETATE SOLUTION AND ALUMINA CREAM REQUIRED

Sample	ml. lead solution	ml. alumina cream
Raw Cane Sugar	0·5 to 1·0	0
	higher amounts for low pol. sugars	
Raw Beet Sugar	0·5 to 1·0	1

PREPARATION OF BASIC LEAD ACETATE SOLUTION AND DRY LEAD.—The International Commission for Uniform Methods of Sugar Analysis briefly referred to as ICUMSA, has standardised the following method:

A basic lead acetate reagent which complies with the Committee of Analytical Reagents of the American Chemical Society contains basic lead (PbO) not less than 33 per cent. It is dissolved in distilled water to a specific gravity of 1·25 to make the solution known as the "wet lead."

The dry reagent known as "dry lead" used in many countries is prepared by grinding the A.C.S. reagent until 70 per cent passes the 115 Tyler sieve and 100 per cent passes the 35 Tyler sieve.

PREPARATION OF ALUMINA CREAM

A cold saturated solution of potash alum in distilled water is prepared and to it ammonium hydroxide (S.G. 0·880) is added until the solution is alkaline. After allowing the precipitate to settle in a tall glass jar the supernatant solution is decanted away and the precipitate washed by decantation until the wash water is practically free from sulphates. Excess water is poured off and the reagent stored in stoppered bottles for use.

Fig. 1 shows a saccharimeter with sodium lamp illumination, a B.S. pol. flask, nickel dish and stirrer, and the operation of pouring on to the pleated filter. Also seen are the sample bottles of raw sugar and a brass tube sampler, "The Connor Corer," which is used to remove an approximation to the normal weight by plunging it vertically into the bottle of sugar after first removing the top inch, in case alteration of moisture content has taken place.
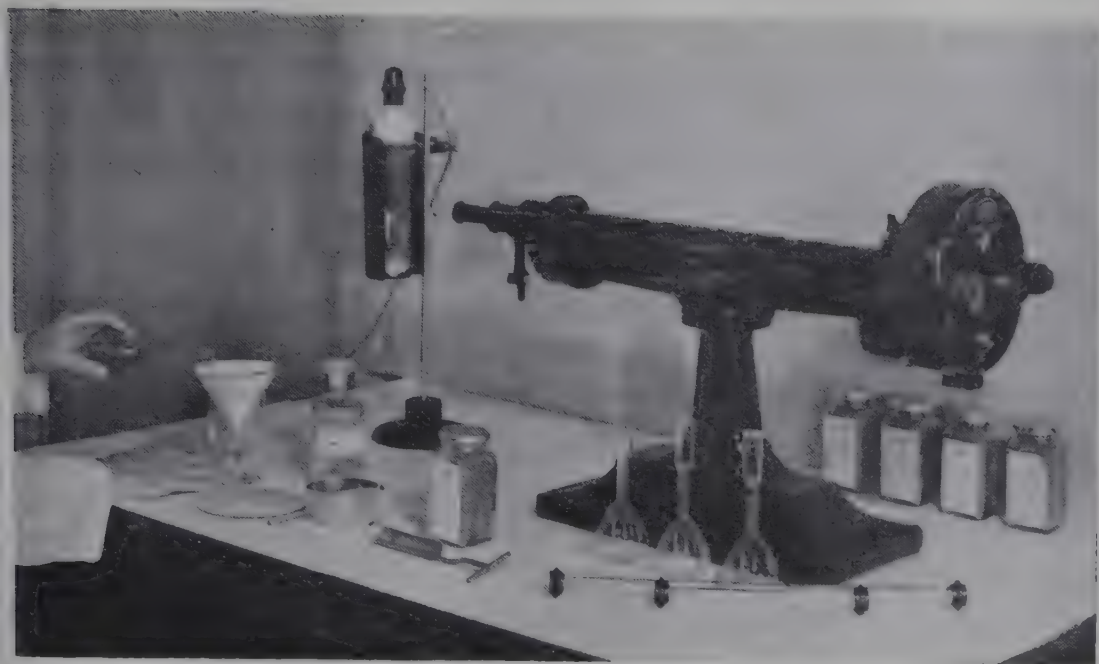
FIG. 1.—POLARIMETER WITH SODIUM LAMP ILLUMINATION.

Raw sugar samples, B.S.I. Pol. Flasks.   Pol. Tubes 200 and 400 mm. Connor corer, metal dish, filtration of solution.

All polarisation operations are conducted at 20° C. and the saccharimeter should be housed in a constant temperature room or at least a constant temperature cabinet.

2. Reducing Sugars

Since it was first shown that some sugars were capable of reduction of copper in a heated alkaline solution and the well-known Fehling's solution of Soxhlet containing copper sulphate, caustic soda and Rochelle salt was formulated, many modifications of the method have been devised and many alternative reducing methods have been proposed, some of which are in current use in other countries.   One of the most well known of the modified Fehling's solution methods which has had acceptance by many official bodies and which is most generally used in this country is the method of Lane & Eynon.

In the determination of reducing sugars by direct titration against Fehling's solution it had originally been assumed that concentration x of the sugar solution multiplied by the volume y, required for complete reduction of a fixed quantity of copper solution was constant, viz. $xy = K$.   This was later disproved. Lane & Eynon still later produced a table of factors to obviate the error and also used an internal indicator, methylene blue solution added towards the end of the titration to enhance the

final end point. They also introduced correction tables for reducing effect of sucrose. In order to obviate use of tables it has been shown by Zerban, Hughes and Nygren that although there is no linear relation between x and y, there is however one between the logarithms of x and y.

A simpler method still is the constant volume method in which $xy = K$ for conditions of standardisation, although sucrose effect still needs correction. The constant volume modification of Lane & Eynon for reducing sugars content of raw sugar is the only one described here.

REAGENTS REQUIRED

Soxhlet's Fehling's Solution

(a) 69·28 gm. of crystalline copper sulphate per litre
(b) 346 gm. of Rochelle salt and 100 gm. of caustic soda per litre.
Solutions a and b are mixed in equal volumes and standardised with pure invert sugar solution.

Methylene Blue

0·3 per cent aqueous solution.

STANDARD INVERT SUGAR SOLUTION

23·75 gm. of pure sucrose are dissolved in 120 ml. of distilled water, 9 ml. of conc. hydrochloric acid (S.G. 1·16) is added and the solution after mixing is allowed to stand at room temperature for 8 days and then made up to 250 ml. The 10 per cent invert solution thus made is further diluted. 200 ml. with addition of N. sodium hydroxide solution added slowly with swirling to mix so that when further diluted to 2000 ml. it shall have an acidity of 0·001 N. expressed as HC1. Alkaline invert sugar solutions quickly decompose. If 4 gm. benzoic acid is added before final dilution the solution can be kept in stock for several years without change, alternatively it is stable for several months.

Standardisation of Fehling's Solution

10 ml. of the Fehling's solution is boiled in a conical flask on a Vitreosil plate over a bunsen burner, with slightly less than required standard invert solution (added from a burette) plus sufficient distilled water to make volume to approximately 60 ml. and addition of 0·5 ml. of 0·3 per cent methylene blue solution. Further small additions of invert solution are made until the methylene blue colour is just discharged.

A further test is made with a closer first approximation to requirements and further small addition of invert sugar solution until the end point is just reached.

The invert equivalent of 10 ml. of Fehling's solution is thus determined.

DETERMINATION OF REDUCING SUGARS CONTENT (AS INVERT) IN RAW CANE SUGAR

The same procedure is followed using 10 ml. Fehling's and in this case a solution of raw sugar containing 10 gm. per 100 ml. and the reducing sugars content in the volume required to reduce 10 ml. Fehling's is calculated to percentage reducing sugar in the raw sugar.

In this case a correction for the reducing effect of sucrose is required and can be obtained from Maltby's Table shown below. Apparent reducing sugars must be multiplied by the factor corresponding to sucrose present to give true reducing sugar content.

Gm. sucrose present in reac-tion flask	.	.	.	0	0·5	2·5	5·0	12·5
Factor	.	.	.	.	0	0·97	0·91	0·86	0·78

Fig. 2 shows the apparatus used for reducing sugar determination. The housing round the burner is necessary to prevent draughts affecting rate of heating and transparent silica plate enable the progress of the reaction to be viewed satisfactorily.
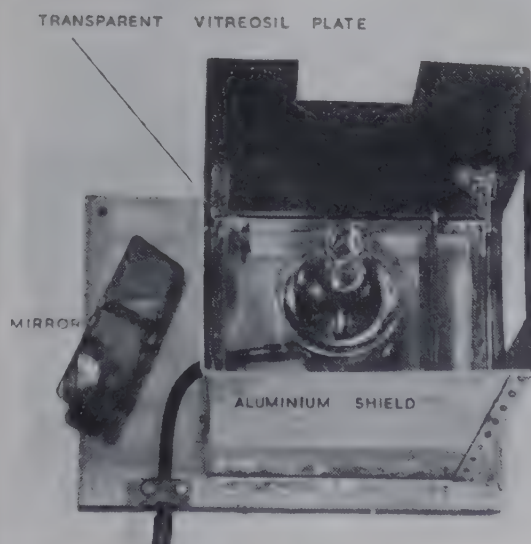
FIG. 2(a) (*Left*).—APPARATUS USED FOR REDUCING SUGAR DETERMINATION.
(b) (*Right*).—THE APPARATUS SEEN FROM ABOVE.

Housing is designed to shield from draughts and illumination is schemed to facilitate exact end point determination.

M

3. Moisture

Raw sugar samples are received in sealed glass bottles. If they represent bulk deliveries any lumps in the sugar have been broken up before bottling. The top one inch of the sample is discarded if not already used for previous tests and no further treatment of the raw sugar is made.

Using the corer, stab samples are made to provide 10 grams which is weighed into a tared flat aluminium dish provided with a close fitting lid (*see* Fig. 3).
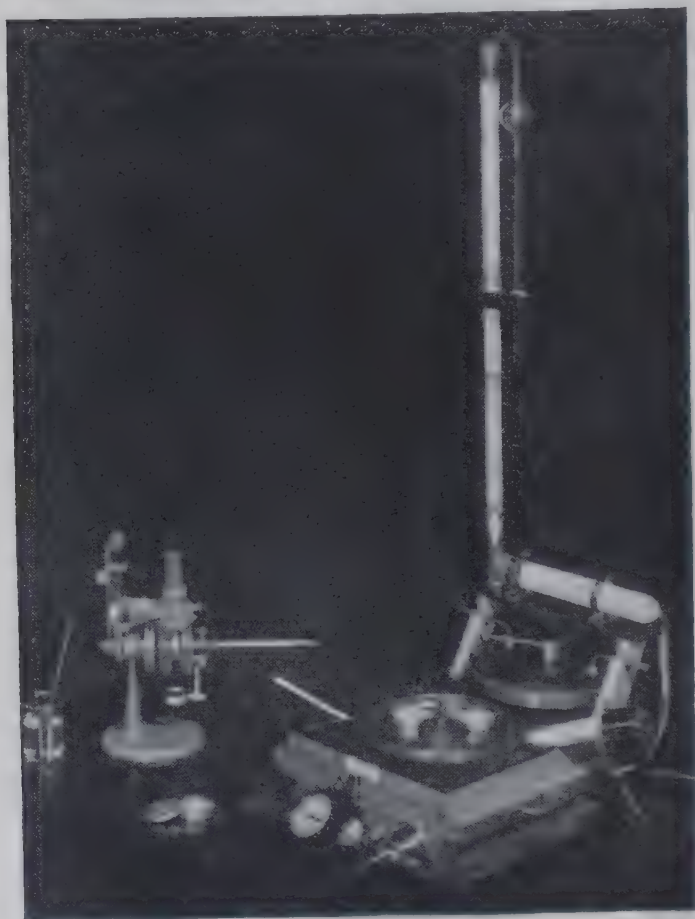
FIG. 3.—GARDINER OVEN, OPEN SHOWING ALUMINIUM DISHES AND ZEISS ABBÉ REFRACTO-METER.

The dish is placed in an electrically heated oven at a temperature of 105° C. $\pm$ 0·5°. The thermometer should nearly reach the shelf holding the dishes and the latter should not be near to the hotter walls of the oven. After 3 hours drying the dish is transferred to a sulphuric acid desiccator containing a thick slab of copper to facilitate cooling. The dish is then reweighed. Loss is taken as moisture and is calculated to percentage moisture. Vacuum drying methods at lower temperatures are too slow and unsuitable for practical usage although they are less destructive of reducing sugars and are used to standardise more rapid methods at higher temperatures. Fig. 3 shows the Gardiner Oven which utilises uniform heating and provides a current of

pre-dried air over the dishes. It can be used over a range of temperatures both with or without vacuum and was specially designed for drying molasses samples diluted with water and tended to give maximum drying surface on aluminium powder. A similar method of treatment is used for syrupy raw sugars in this or the ordinary oven.

4. Ash

Approximately 5 grams of raw sugar are weighed into a platinum dish and moistened with drops of concentrated sulphuric acid equivalent to a volume of 0·5 ml., gentle heat being applied by a bunsen burner with swirling of the dish to aid mixing. As soon as the sample has become carbonised the dish is placed in an electric or gas muffle furnace (temperature approximately that indicated by a pyrometer as 550° C.) until the ash is apparently free from carbon. The ash is re-sulphated by moistening with a few drops of concentrated sulphuric acid and dish replaced in the muffle now at 800° C. Dish and contents are re-heated until weight is constant. From the weight of residual ash the percentage of sulphated ash is calculated.

It was formerly customary to deduct $\frac{1}{10}$ of the weight of sulphated ash and report result as gravimetric ash. Some countries calculate nett rendement of yield or refined sugar from raw sugar by multiplying gravimetric ash by 5 and deduct this from polarisation.

If the sulphated ash figure is used a factor of 4·5 is required to give the same nett rendement.

5. Sucrose

The polarisation of raw cane sugar requires a correction for the lead error caused by defecation with basic lead acetate.

This error is due to three major effects: (a) volume of precipitate; (b) lead levulosate; (c) effect on rotation of sucrose, and Table I, accepted by the British National Committee of ICUMSA, has to be used to obtain the necessary deduction to be made from the observed reading.

The corrected pol. is even then only an approximation to the true sucrose content. Sucrose can be determined by the Clerget method by polarising before and after inversion but there are several objections to this method. It is lengthy and also involves experimental errors approaching the order of the difference between pol. and sucrose. A more practical method used in our refineries is to correct for the polarising effect of the reducing

TABLE I

Observed Pol. of Sugar	Error per ml. of basic lead acetate solution used in polarisation
95·0	0·167
·2	0·161
·4	0·155
·6	0·149
·8	0·143
96·0	0·137
·2	0·131
·4	0·125
·6	0·119
·8	0·113
97·0	0·107
·2	0·101
·4	0·094
·6	0·088
·8	0·082
98·0	0·076
·2	0·070
·4	0·064
·6	0·058
·8	0·052
99·0	0·046
·2	0·040
·4	0·034
·6	0·028
·8	0·022
100·0	0·015

sugars and other non-sugars by addition to the pol. of $0.22 \times$ per-centage reducing sugars.

$$S = \text{Pol.} \times 0.22 \text{ I}$$

Theoretically the correction for invert sugar would require a factor of 0·30 but the reducing sugars are never present in exactly equi-molecular proportions and non-sugars also influence the polarisation.

6. Non-Sugars

The sum total of sucrose, reducing sugars, moisture, gravimetric ash, deducted from 100 gives the content of organic non-sugars.

If polarisation is used instead of sucrose the sum total deducted from 100 gives the balance usually termed "unestimated."

MOLASSES

1. Total Sugars
2. Water by Refractometer
3. Ash
4. Density or weight per cubic foot

1. Total Sugars

In this country the British National Committee of The International Commission for Uniform Methods of Sugar Analysis have recommended the chemical method instead of the optical method of Clerget and it is adopted by our refineries.

Hot Invertase Inversion and Total Sugars after Inversion

5 grams of molasses are washed into a 100 ml. flask, 8 drops of glacial acetic acid (pre-determined to make pH 4·7) are added and 0·5 to 1·0 ml. of "Sumasuco" invertase added; the solution of about 80 ml. is heated for 15 minutes at 55° C. to 60° C. for inversion of the sucrose. The amount of invertase required is dependent on the concentration of sucrose in the solution, and can be determined by a trial with pure sucrose and slight excess provided (correction for reducing effect of invertase may be necessary). After inversion the solution is rapidly cooled; dilution is made by washing into a 1 litre flask and making volume of mixed contents about 500 ml. before adding about 10 ml. of 5 per cent potassium oxalate solution (actual volume is pre-determined to give a slight excess of oxalate). It is then made to the mark, mixed, filtered and used for titration against 25 ml. of Fehling's solution as already described under raw sugar. The Fehling's solution being standardised in the same manner as the test. Correction is made for sucrose present in the test as shown in the section on raw sugars.

Direct Reducing Sugars

The quantity weighed is dependent on the percentage of reducing sugars present. With cane molasses, 10 grams of the sample are weighed, dissolved, transferred to a 1 litre flask and a pre-determined volume of potassium oxalate solution added. The volume is then made to mark, filtered and used for titration against 10 ml. of Fehling's solution.

Correction is made for sucrose effect as with raw sugars.

Reducing Sugars after Inversion — Direct Reducing Sugars = Sucrose inverted and now present as Invert Sugar.

$$\text{Invert Sugar} \times \frac{19}{20} = \text{Sucrose.}$$

Sucrose + Direct Reducing Sugars = Total Sugars.

2. Water by Refractometer

The International Scale of Refractive Indices of Sucrose Solutions (ICUMSA Proceedings 1936) relates Refractive Index @ 20° C. to percentage Sucrose in solution. Readings are made with illumination by sodium D light but white light or a tungsten filament lamp are customarily used.

As molasses, both beet and cane, contain soluble matter other than sucrose, the direct refractive index only approximates to the sucrose content. Corrections are necessary for the percentage of reducing sugars present and also for non-sugars. The latter correction is based on sulphated ash content.

If the molasses contains undissolved sugar, a 10 per cent dilution (not solution) is made by addition of a weighed amount of water to a weighed amount of sample and all sugar dissolved before transferring a drop to prism of the Refractometer. Fig. 4 shows a Zeiss Sugar
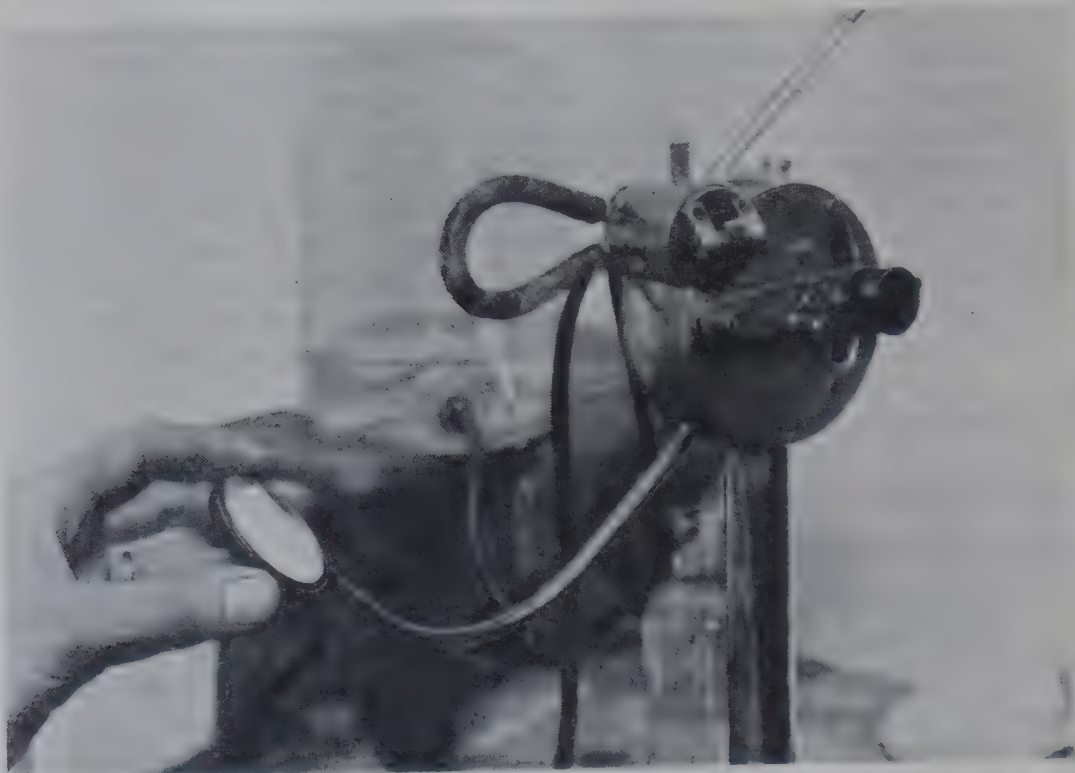
FIG. 4.—ZEISS SUGAR SCALE REFRACTOMETER USING REFLECTED LIGHT FOR MOLASSES.

Scale Refractometer which is graduated in °Sugar. This instrument is provided with an alternative illumination for reflected light particularly suitable for dark solutions like molasses.

The prisms are jacketed so that a stream of water can be run through to enable readings to be taken at 20° C. The refractive index at 20° C. gives equivalent sucrose content from Table II.

Having found this, correct if necessary for 10 per cent dilution, by dividing result by 0·9.

When ash content is low and with the reducing sugars and approximate solids (sucrose) known, the correction for reducing sugars (y) can be calculated from the equation

$$y = 0·00025 \, x \quad \text{reducing sugars where } x = \text{true solids.}$$

In the case of a large correction (high content of reducing sugars) the observed solids are used to obtain approximate correction and this is added to observed solids and the exact correction then calculated.

For high ash content the correction based both on sulphate ash and on reducing sugars is either

(a) True Solids of cane molasses $= \dfrac{\text{refractometer solids (sucrose table)}}{1 - 0·00025 \text{ reducing sugars} + 0·0043 \text{ sulphate ash}}$

(b) True Solids of beet molasses $= \dfrac{\text{refractometer solids (sucrose table)}}{1 - 0·00025 \text{ reducing sugars} - 0·0008 \text{ sulphate ash}}$

3.   Ash

3 grams of molasses is weighed into a platinum dish and 0·5 to 1·0 ml. of concentrated sulphuric acid added and the method used for raw sugar ash followed.

4.   Density or Weight Per Cubic Foot.

The Brix Hydrometer is graduated in percentage sucrose for an air free solution when read at 20° C. The °Brix (°Bx) reading does not give a correct measure of dissolved solids in sugar solutions of low purity such as molasses because the non-sugars have an affect on the density which differs from that of sucrose. For such solutions the °Bx is nevertheless a useful means of determining the density and it is used for gauging the weight of molasses in tanks and barges. Such viscous material as molasses may contain a large amount of occluded air. It is necessary to know the apparent specific gravity if this is to be used to calculate the weight of the molasses from the measured volume obtained

TABLE II

Sucrose per cent	$20 \atop nD$	Sucrose per cent	$20 \atop nD$
0	1·33299	43	1·4056
1	1·33443	44	1·4076
2	1·33588	45	1·4096
3	1·33733	46	1·4117
4	1·33800	47	1·4137
5	1·34027	48	1·4158
6	1·34176	49	1·4179
7	1·34326	50	1·4200
8	1·34477	51	1·4221
9	1·34629	52	1·4242
10	1·34783	53	1·4264
11	1·34937	54	1·4285
12	1·35093	55	1·4307
13	1·35250	56	1·4329
14	1·35408	57	1·4351
15	1·35567	58	1·4373
16	1·35728	59	1·4396
17	1·35890	60	1·4418
18	1·36053	61	1·4441
19	1·36218	62	1·4464
20	1·36384	63	1·4486
21	1·36551	64	1·4509
22	1·36719	65	1·4532
23	1·36888	66	1·4555
24	1·37059	67	1·4579
25	1·3723	68	1·4603
26	1·3740	69	1·4627
27	1·3758	70	1·4651
28	1·3775	71	1·4676
29	1·3793	72	1·4700
30	1·3811	73	1·4725
31	1·3829	74	1·4749
32	1·3847	75	1·4774
33	1·3865	76	1·4799
34	1·3883	77	1·4825
35	1·3902	78	1·4850
36	1·3920	79	1·4876
37	1·3939	80	1·4901
38	1·3958	81	1·4927
39	1·3978	82	1·4954
40	1·3997	83	1·4980
41	1·4016	84	1·5007
42	1·4036	85	1·5033

by a graduated dip rod and the tank area. Fig. 5 shows the relation between °Bx and specific gravity.
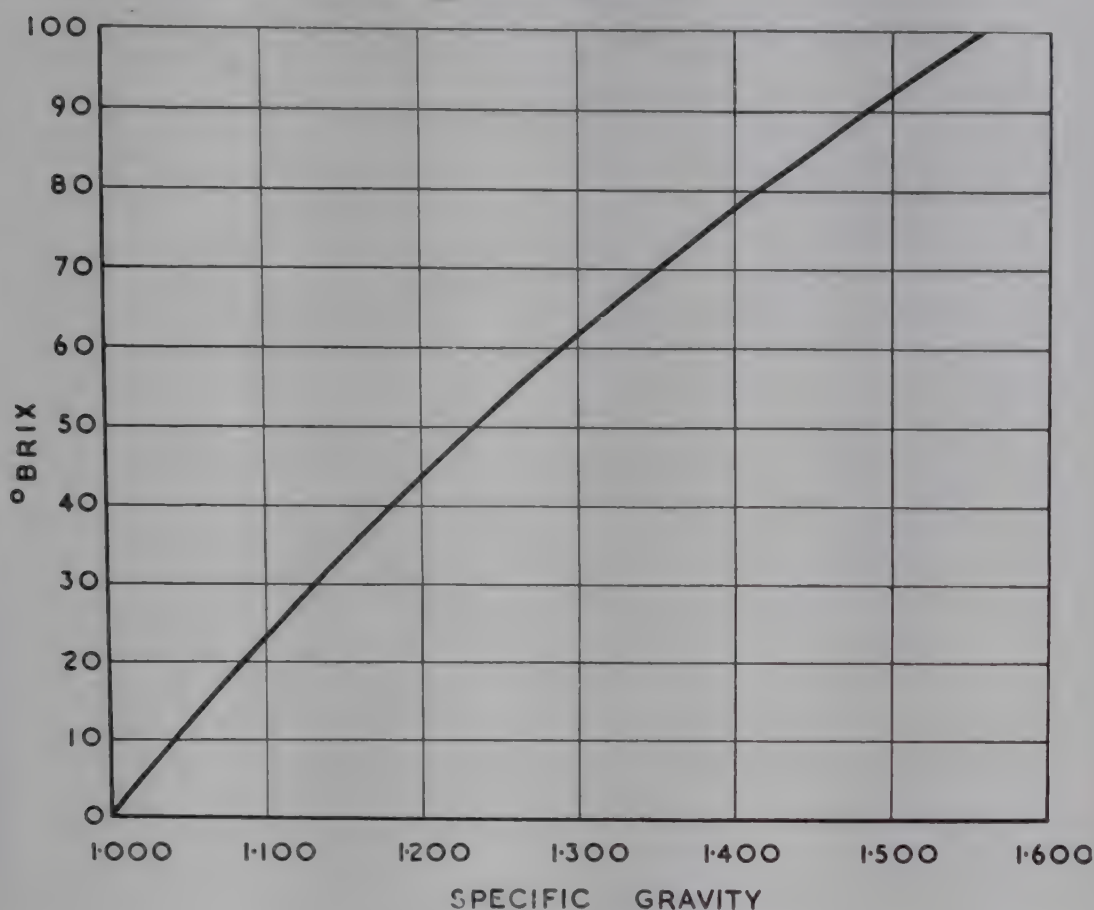
FIG. 5.—THE RELATION BETWEEN °Bx AND SPECIFIC GRAVITY.

To determine the °Bx, a sample of the molasses is carefully poured into a glass hydrometer jar holding this in a sloping position to avoid trapping any more air. When nearly filled a suitable Brix Hydrometer is selected from the range of hydrometers available and is carefully inserted into the molasses. When it has settled to its equilibrium position, the meniscus level is read on the hydrometer and the temperature read by inserting a chemical thermometer with the bulb in proximity to the centre of the immersed portion of the hydrometer. The °Bx reading can be approximately corrected for temperature by subtracting 0·9 for every 10° C. reading below 20° C. and adding 0·9 for every 10° C. above 20° C. For more exact work, physical tables of temperature corrections must be consulted.

OTHER CONTROL TESTS

1. Sulphur Dioxide

This compound free or combined as sulphite is used as a preservative in a restricted number of foodstuffs, notably fruit pulp for jam, dried fruits and some beverages. It is also used as

a hydrolytic agent in glucose manufacture and as a bleaching agent in preparation of crystallised fruits. Public Health regulations of 1925 limited the amount of SO_2 allowed in various foods and for this reason its content in sugar and sugar products is of importance, less because amounts present might exceed statutory limits in sugar and sugar products sold for direct consumption but more because any such products used in manufacture might contribute to the total SO_2 in such manufactures.

Sulphur dioxide is used in the sulphitation process in preparation of some raw cane and raw beet sugars and also for plantation or factory white sugars. Very little or none is used in refineries but residual SO_2 may be present in raw sugars received for refining and although after refinement white sugars contain only 1 or 2 parts per million, some lower purity final products through concentration due to elimination of some sucrose, contain larger amounts. SO_2 determination is therefore a necessary part of analytical control in the refinery both for raw sugars and final products.

The method used that of the B.A.R., now called the British Food Manufacturing Industries Research Association, consists of distilling a solution of the sample with phosphoric acid collecting the condensed distillate in standard iodine solution which is being titrated against standard thiosulphate using starch as indicator whilst the distillation is proceeding. The chief advantages of the method are its rapidity and the prevention of loss by oxidation.

A double surface (Davis) condenser is preferable.

FIG. 6.—
APPARATUS FOR
DETERMINATION
OF SO_2.

SO₂ METHOD

Reagents required are:

(1) 20 per cent phosphoric acid solution (28° Bx)
(2) N/100 iodine
(3) 1 per cent starch
(4) 50° Bx sugar solution containing known additions of sulphite ($\cdot$03 gm. SO_2 per litre).

The *Apparatus* is shown in Fig. 6.

It consists of a round bottom flask fitted with tap funnel and leading

tube carrying the B.A.R. still head from which distillate freed from splashes passes through a vertical Liebig condenser and terminates in a B.A.R. adapter of the bubbler type which dips into the starch-iodine solution contained in the beaker.

PROCEDURE

200 ml. distilled water, 50 ml. of the sugar solution and 25 ml. phosphoric acid reagent are added to the flask after assembly of the apparatus. Sufficient distilled water is placed in the beaker to cover the bell of the absorption adapter. A few ml. of starch solution are also added to the beaker. The flask is heated with the naked flame of a large burner to bring contents to the boil in $2\frac{1}{2}$ minutes. Condensate + water in the beaker should not reach a temperature above 27° C. Iodine solution is run in from a burette to react with liberated SO_2.

The preliminary distillation of about 3 minutes gives results lower than theoretical. After the first test without alteration to the apparatus another 50 ml. of the sugar solution is quickly added and re-distilled and the SO_2 found approximates closely to theory.

1 ml. N/100 Iodine solution = ·00032 gm. SO_2

$$\frac{\text{ml. iodine solution} \times \cdot 00032 \times 10^6}{\text{gm sample in sugar solution used}} = \text{parts } SO_2 \text{ per million of sample}$$

2. Arsenic

Arsenic is not a normal constituent of sugar and other food materials but may have been introduced by treatment with impure chemicals in preparation of raw sugar. As arsenical compounds are used as sprays for killing blights and insects and also against locusts, all food materials need examination to see that they are free from this exceedingly dangerous element.

ROYAL COMMISSION ON ARSENICAL POISONING.—A number of cases of fatal poisoning with arsenic were traced to arsenical pyrites in manufacture of sulphuric acid. This acid was used for inversion of sugar in preparation of brewers' sugar, or saccharum, and this, as its name suggests, was introduced into beer.

As a result of this the Royal Commission on Arsenical Poisoning was appointed and in their report in 1903, recommended that no substances used in the manufacture of food or drink should contain more arsenic than 1/100 grain per lb. (= 1·4 parts per million) of arsenic as As_2O_3. A more recent order has confirmed the limit as 1 part As. per million.

SAMPLES TESTED FOR ARSENIC IN SUGAR REFINERY LABORA-
TORIES.—Every different mark of raw sugar (representing
different raw factories) in every ship load received at the refinery
is tested for traces of arsenic. All chemicals used in the refinery
are likewise tested whether they are used in the refining process
or not.

Paints, tiles, cements, metals, packings, jointings, cardboard,
paper, and printing inks, etc., are all examined to safeguard
against contamination of the refined sugar and other finished
products.

METHOD OF DETERMINATION OF TRACES OF ARSENIC.—Three
methods are commonly used. They are the Gutzeit, the electro-
lytic method, and the Marsh-Berzelius.

The last, described below, is a derivation of the familiar Marsh
test of qualitative analysis.

Arsenic compounds are reduced by nascent hydrogen generated by
the action of acid on zinc as shown in the following equations:
$$As_4O_6 + 12 H_2 = 4AsH_3 + 6H_2O$$
$$4AsH_3 = 4As + 6H_2$$
The arseniuretted hydrogen is decomposed to elemental arsenic, which
forms as a mirror or stain, by passing it through a heated hard glass
tube.
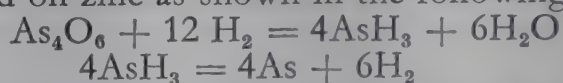
APPARATUS REQUIRED FOR THE MARSH-BERZELIUS TEST
This is shown in outline in Fig. 7.

It consists of a glass flask (200 cc.) half filled with granulated zinc and
provided with a cork fitted with a thistle funnel and a tube bent at
right angles leading to two wider corked tubes. The first tube con-
tains lead acetate paper and cotton wool plugs at each end. The
second tube contains granular calcium chloride and cotton wool plugs
at each end. A further tube is connected to the hard glass arsenic
tube which is drawn out into a thin capillary and bent as shown in
Fig. 7.

REAGENTS NECESSARY.—The purity of these and their freedom
from any traces of arsenic is a *sine qua non*.

1. Granulated zinc, arsenic free.
2. Hydrochloric acid solution, 20 per cent HCl, arsenic free.
3. Lead acetate paper prepared by immersing strips of sheet filter
   paper in 12 per cent lead acetate solution (neutral in reaction). It
   is dried at atmospheric temperature out of contact with H_2S.
4. Cotton wool, absorbant quality, is also soaked in similar lead
   acetate solution and dried in a similar way to the paper.
5. Cadmium sulphate. Pure crystalline salt (arsenic free) made to a
   6% solution in distilled water.
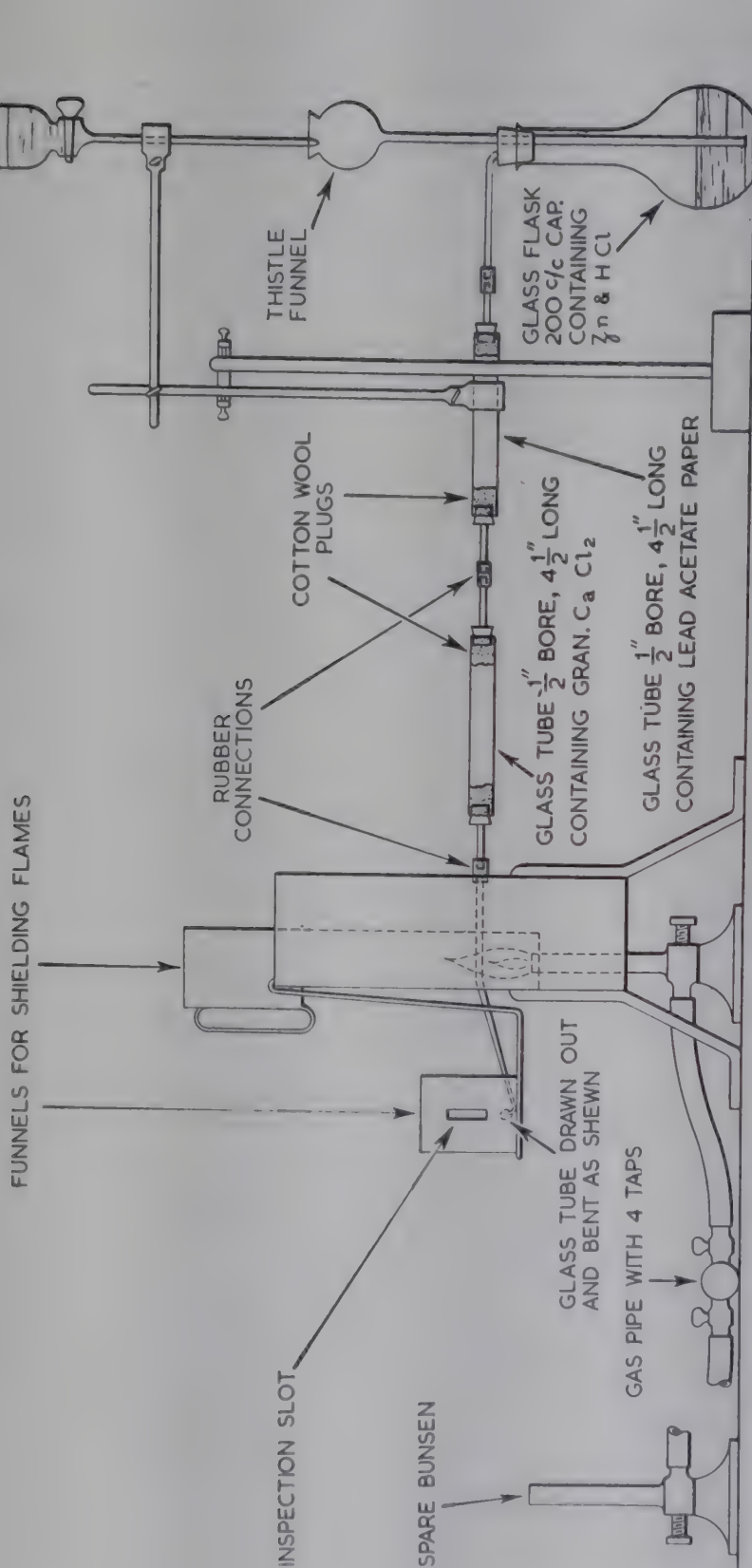
Fig. 7.—Apparatus Required for the Marsh-Berzelius Test for Estimation of Arsenic.

Procedure

Calcium chloride, cotton wool and lead acetate papers are examined to see that they are quite dry and clean.

One tube is filled with calcium chloride (Fig. 8) and the other with the lead acetate paper.   The arsenic tube is connected with its shoulder just inside the metal shield (Fig. 9) in the place where the flame will heat it.   Next the hydrogen generating flask is connected and seven drops of cadmium sulphate solution added to increase the sensitivity of the zinc, and distilled water to just cover the zinc.   Acid is added in sufficient quantity to promote fairly vigorous evolution of hydrogen. The apparatus is tested for leaks by applying finger over jet.   If quite tight, acid is forced up into the thistle funnel.   The finger is removed and hydrogen allowed to stream through the apparatus for 3 to 4 minutes to displace all air from apparatus.   The jet can then, *and only then*, be lighted safely.

When the first large hydrogen flame has abated somewhat the bunsen burner is lighted and also the spare burner which is used to drive off any condensed moisture in the arsenic tube.   The shield is then adjusted to keep off draughts.

Blank Test

This is to check purity of reagents.   A solution of 20 grams of pure sugar in a little distilled water to which 2 ml. of HCl solution have been added is poured into the thistle funnel (Fig. 10) and the flow of hydrogen, as shown by the flame at the jet, is controlled by the rate of addition of acid from the tap funnel.

The flow of hydrogen is kept constant for 20 minutes and if reagents are quite free from arsenic no stain is obtained in the tube (Fig. 11).

Raw Sugar Sample

20 grams of this having been weighed out in a small beaker and dissolved in a little distilled water and HCl as before, is then poured in the thistle funnel and the same procedure followed for another 20 minutes.   The resultant stain, if any, is then compared with standard stains and the arsenic content thus assessed.

Organic Matter Destruction

It is to be noted that the sugar has not to be destroyed before arsenic can be estimated by the Marsh-Berzelius method.

With some foodstuffs, e.g., flour, the organic matter must be oxydised first by heating with sulphuric and nitric acid (the wet method) or by heating with dry magnesium carbonate (the dry method).

The products then obtained are treated with HCl and then introduced into the Marsh-Berzelius apparatus as already described.

Presence of Antimony

Antimony compounds are reduced in the Marsh-Berzelius test and give a stain resembling that of arsenic.

FIG. 8.—THE MARSH
BEZELIUS TEST FOR
ARSENIC.

Granular calcium chlor-
ide being placed in the
second tube. Lead acetate
paper is shown in the first
tube, granulated zinc in the
flask and HCl solution in
the tap funnel above.

FIG. 9.—THE MARSH BERZELIUS TEST FOR ARSENIC.
The arsenic tube with shoulder in metal shield is seen on the left and immed-
iately above the burner. *Right*, the zinc is being covered with distilled water.

FIG. 10.—THE MARSH BERZELIUS TEST FOR ARSENIC.

Adding the sugar solution to the flask via the thistle funnel. The glass rod should reach from clip of beaker to interior side of funnel.

FIG. 11.—THE MARSH BERZELIUS TEST FOR ARSENIC.

Results of test. The glass tubes are cut and the central portion mounted on card for record purposes. Blank tests alternating with sensitive tests showing a part per million are shown on the card.
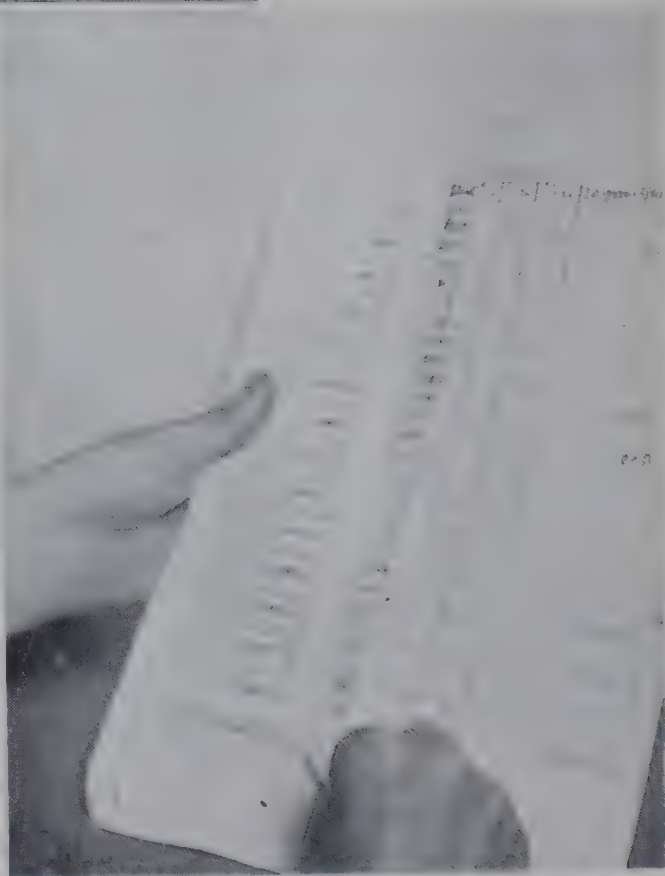
This can, however, be readily distinguished because it is insoluble in dilute sodium hypochlorite solution whereas the stain due to arsenic is readily soluble.

3. Lead

A recent statutory order of the Ministry of Food has imposed limits on lead content of foods. These limits are in some cases much lower than previously considered necessary or even possible in the food industry. After much consultation with manufacturers and after long consideration of evidence, a general limit of 2 p.p.m. lead in foods has been fixed with the exception of some scheduled foods which have higher or lower limits. Sugar and sugar products are partly included in the schedule.

Refined white sugar (sulphated ash content not exceeding 0·03 per cent). } 0·5 p.p.m.

All types of sugar, sugar syrups, invert sugar and direct consumption coloured sugars (sulphated ash content exceeding 1·0 per cent).
Raw sugars for refining.
Edible molasses. } 5·0 p.p.m.

This leaves unscheduled, all other sugar products (sulphated ash between 0·03 and 1·0 per cent), also raw sugars used for direct consumption or for manufacturing purposes. } 2·0 p.p.m.

In a recent investigation in British West Indies cane factories, the writer has shown that sugar cane reaching the factory contains variable small amounts of lead. In the factory variable amounts are picked up in the process of producing raw or coloured sugars but elimination in defecation, and scaling of evaporators usually compensates for this pick up. The sugars and molasses produced were all within the limits of the Lead Order. Nevertheless as raw sugars for British Refineries come from many different countries it is necessary to test raw sugars and finished products to ensure that limits of lead are not exceeded.

Method of Determination of Lead

Reagents

1. Ammonium thiocyanate, saturated: 160 grams of the A.R. salt + 100 ml. water (= 225 ml. solution), filtered.
2. Hydrochloric acid 1:1. 1 volume of concentrated HCl AR + 1 volume water.

N

3. Mixed solvent: equal volumes of amyl alcohol A.R. and ether A.R.

4. Ammonium citrate 50 per cent W/V: 186 grams citric acid A.R. + 0·88 ammonia solution A.R. until neutral to litmus. Solutions diluted to 400 ml. and filtered.

5. Ammonia solution: 0·88 sp. gr. A.R.

6. Potassium cyanide: 10 per cent W/V of the A.R. salt.

7. Sodium sulphide: 10 per cent W/V of $Na_2S. 9 H_2O$. A.R.

8. Standard lead solution: (a) 0·160 gms. $Pb (NO_3)_2$. A.R. + 0·5 ml. conc. HNO_3, diss. to 100 ml.; (b) 1·00 ml. of solution (a) diluted to 100·0 ml.
   1 ml. of (b) = 0·01 m. gm. Pb.
   Solution (b) may not keep very well.

9. Magnesium acetate: 20 per cent W/V of $(CH_3COO_2)_2$ Mg. $4H_2O$ A.R.

10. Sulphuric acid concentrate: "B.D.H. for Foodstuff Analysis" Pb content $\not> 0·005$ p.p.m.

11. Water: All double-distilled, from Pyrex still.

PROCEDURE

For most samples the equivalent of 10 grams of solids is weighed. In the case of molasses, 3 to 4 grams is sufficient. The sample is weighed into a platinum (or silica) dish, water added to dilute to ca. 70° Bx, then 20 to 30 drops of concentrated sulphuric acid added, mixed, and dish heated on water-bath until contents have nearly solidified. Residue is gently carbonised over small flame, then dish is transferred to muffle at ca. 250° C., and temperature is raised slowly to 450° C. to 500° C. The temperature should not exceed 500° C. at any time. Ashing is continued until all carbon has been oxidised. Samples which give a little persistent carbon, appearing as a greyish ash, may often be improved by "fuming" with a few drops of concentrated sulphuric acid in another crucible. Re-sulphation of the ash is more effective, but should not be used with samples having a high calcium content.

The final ash is treated with 5 ml. of 1:1 hydrochloric acid + 2 to 3 ml. of water, covered with clock glass and heated on water-bath for 10 to 15 minutes. The solution is cooled, transferred to a 50 ml. stoppered separating funnel with two 4 ml. washings with water, and a few drops of N/10 potassium permanganate added (enough to give a pink colouration lasting for a few seconds) to oxidise iron. 4 ml. of ammonium thiocyanate (saturated) is now added, followed by 20 ml. of mixed solvent. The funnel is shaken, and the aqueous layer which separates it transferred to another funnel. 15 ml. of solvent is added, and solution again extracted. Usually two extractions are sufficient to remove the whole of the ferric thiocyanate, but if much is present, a third extraction may be required. The final aqueous layer, if not perfectly clear, is filtered into a 50 ml. beaker using an acid-rinsed 7 cm. paper (Whatman 44). To the filtrate is added 3 ml. of 50 per cent ammonium citrate (Note 3), ammonia solution until neutral to litmus

then 0·5 ml. in excess, followed by 1·0 ml. of 10 per cent potassium cyanide. A solution for balancing is prepared at the same time by adding to another beaker similar volumes of citrate, ammonia and cyanide. For the colorimetric comparison, tall, narrow 50 ml. tubes are desirable, having a bore of 14 mm., which gives a column of ca. 30 cm. (Note 4). The two solutions are transferred to such tubes and compared. The balancing solution should be quite colourless. If the solution of sample has a slight off-tint, this is first balanced by adding to the other tube 1·0 ml. of sodium sulphide, then standard lead solution (*b*) from burette until in mixing the depth of off-tint of sample is matched. The volume of lead solution required is noted. Sulphide is now added to the solution of sample and the colouration produced again matched by adding lead solution to the balancing solution. The total volume of lead solution required (Vol. "A") minus that needed for balancing off-tint gives the equivalent of lead in sample taken. (Note: The depth of colouration resulting when lead solution is added after the addition of sulphide is not always the same as when it is added before the sulphide. It is therefore advisable when the approximate lead equivalent has been obtained to repeat the balancing, adding the bulk of the standard lead solution before the sulphide is added.)

A "blank" determination should be run, working on 0·3 gm. pure sucrose and using the same volume of reagents and same technique as for sample. The figure obtained (ml. of standard lead solution) is deducted, together with that for off-tint, from volume "A."

Note 1. When ash content of material is < 0·5 per cent on solids it is desirable to add 2 ml. of 20 per cent magnesium acetate to sample before ashing. Samples thus treated seem to give a final solution of greater off-tint. This can be overcome by re-sulphation of the ash.

Note 2. All glassware used (including reagent bottles) should preferably be of "Pyrex."

Note 3. The volume of ammonium citrate should be increased for samples which have a high content of Mg. or Ca., in order to avoid possible precipitation later.

Note 4. Comparison tubes. These are made up from soda-glass tubing (14 mm. bore) and 2 in. × $\frac{1}{16}$ in. glass circles (from J. Hetly & Co. Ltd., 35, Soho Square, London, W.1), joined with Araldite Cement, type 1.

H. C. S. de W.

CHAPTER 14

JAMS

THE production of a uniform high quality jam and the avoidance of failures from such variable raw materials as fruit, demands a large measure of chemical control. The methods of control, however, must be quick so that if some correcting action is required, it can be taken before a large quantity of substandard jam is produced.

The most important determinations to be made are:

1. Soluble Solids Content
2. Invert Sugar Content
3. Hydrogen Ion Concentration
4. SO_2 Content

Soluble Solids Content

This can best be obtained by means of a refractometer. Two types are in common use. The first is the accurate Abbé type which is suitable for use in the laboratory, whilst the second is a large projection type of instrument of special robust design for use in the factory.

THE ABBÉ REFRACTOMETER.—This instrument consists of two prisms between which a few drops of jam are placed by means of a bone or plastic spatula. A plane mirror reflects light upward through the lower prism to the surface of the jam where it is either refracted or totally reflected according to the refractive index of the jam. The refracted light is viewed through a telescope fixed to a sector over which moves an arm attached to the prisms. The sector is usually graduated both in refractive indices and in sugar percentages and the prism arm carries a magnifying lens through which the scale can be viewed. As part of the light is totally reflected, the field of view will be divided into a bright and a dark part. If the prism arm is moved so that the line dividing the bright and dark parts coincides with the crosswire in the eyepiece of the telescope, then the refractive index can be read directly upon the scale. An important feature of the Abbé refractometer is the compensator which corrects the dispersion of the white light passing through the prisms giving a clear cut borderline between the two parts. Refractive indices are usually standardised at 20° C. and correction must be made for any divergence from this temperature. A cooling system is built into the prisms so that the instrument and the thin film of jam can be brought to the same temperature.

Fig. 1.—Abbé Refractometer.

A thin film of jam is placed between the two halves of the prism box. The field is viewed through the telescope and the borderline of shadow made to coincide with the crosswire. The refractive index is then read from the scale.

(*Bellingham & Stanley, Ltd.*)
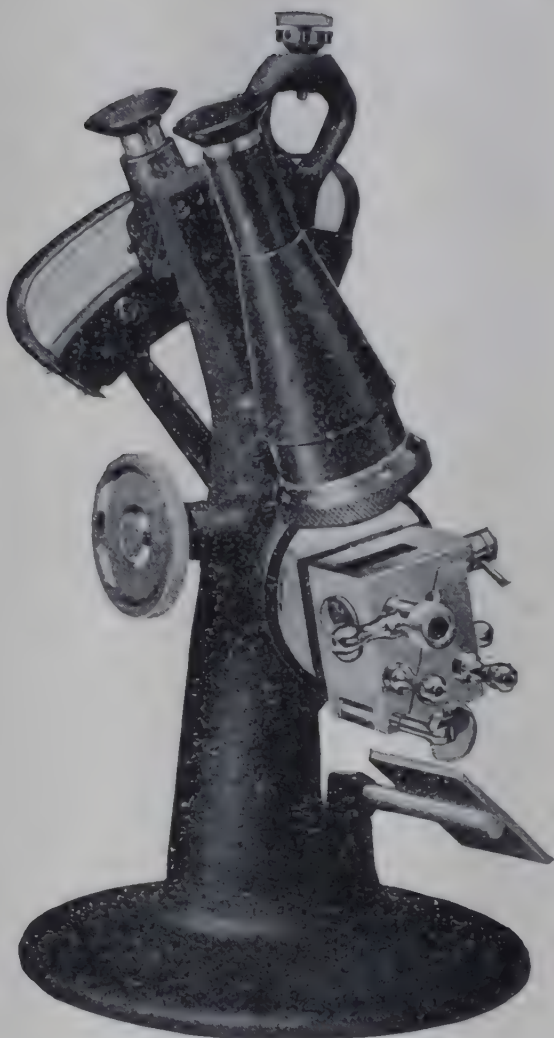
PROJECTION TYPE INSTRUMENT.—This type can be used for rapid determinations at room temperature and is therefore admirably suited for use in the jam-boiling room.

In this instrument, the jam, or sugar solution, is placed on the top surface of the single prism which is illuminated by reflected light from a lamp mounted inside the instrument. The refractive index can then be read on a long scale which is clearly visible with the naked eye.

Fig. 2.—Projection Refractometer.

The sample is spread on the prism at (1) and the refractive index can be read in (2).

(*Bellingham & Stanley, Ltd.*)

The scale is marked in sugar percentages from 30 to 90 and in refractive indices from 1·380 to 1·517. Because the reading depends upon the reflection of a portion of the light entering the prism at the interface between prism and material being tested, the instrument is suitable for use with dark coloured or opaque substances as well as clear sugar solutions.

Sets of tables have been constructed by various authorities showing the relation between refractive index and concentration in sugar solutions. These tables show that solutions of most sugars, including commercial glucose, have refractive indices which differ only very slightly from those of sucrose. Therefore, for practical purposes, the soluble solids in jam can be obtained directly from the refractive index converted into percentage of sucrose, as it is done on the scales of the instruments already described.

Invert Sugar Content Using Fehling's Solution (*see* also p. 168)

Regular and frequent determinations of invert sugar is most important in the control of jam boiling and the results must be made available quickly to enable the jam boiler to make any adjustments in his process that may be necessary. The estimation is made volumetrically with the aid of Fehling's solution, and the method is specially adapted for use with jam. It is advisable to observe closely the details of the method.

FEHLING'S SOLUTION.—This is made up of two solutions which are only mixed together in equal proportions when required.
Solution A.   69·28 gm. Cu SO_4. $5H_2O$ in 1000 ml. of distilled water
Solution B.   346 gm. Rochelle salt   } in 1000 ml. of
              130 gm. Sodium hydroxide   } distilled water.

When required, 25 ml. of each solution, measured in a pipette, are mixed together in a clean dry beaker.

50 gm. of cold jam are weighed out and mixed with about 100 ml. of distilled water in a 300 ml. beaker. The mixture is carefully stirred until all the jam is dispersed in the water and it is then transferred, with washing, to a 250 ml. graduated flask and made up to volume. Very thorough shaking ensures that all the sugars in the jam are brought into solution.

The suspension is then filtered into a conical flask and 25 ml. of the filtrate are pipetted into a 250 ml. flask and made up to volume, giving a 2 per cent solution.

25 ml. of mixed Fehling's solution are pipetted into a 250 ml. conical flask. A burette fitted with a piece of rubber tubing and a

pinch cock instead of a glass tap is filled with the 2 per cent jam solution. 10 to 15 ml. are measured from the burette into the Fehling's solution and then brought to the boil. Keeping the liquid boiling, the jam solution is added, 1 or 2 ml. at a time until the colour of the solution begins to change from blue to red. Three drops of a 1 per cent solution of methylene blue in water are added and the titration continued until the colour turns distinctly red. From the number of mls. of jam solution used, the percentage of invert sugar can be found by use of the table, which is derived from Lane and Eynon's tables.

Greater accuracy can be obtained by repeating the titration. In this second titration, all the test solution required to reach the end point, less 1 ml., are added at once to the Fehling's solution in a conical flask. This is then boiled for precisely 2 minutes; 3 drops of methylene blue solution are added and the titration completed drop by drop. The total boiling time must not exceed 3 minutes. The flask should not be removed from the wire gauze at any stage of the titration and it should not be shaken as the introduction of air will restore the blue colour.

TABLE I

INVERT SUGAR

For a 2 per cent jam solution and using 25 ml. Fehling's solution

Ml. of 2 per cent Jam Solution	Percentage Invert Sugar in Jam	Ml. of 2 per cent Jam Solution	Percentage Invert Sugar in Jam
15	40·80	32	19·20
16	38·30	33	18·67
17	36·00	34	18·13
18	34·00	35	17·60
19	32·30	36	17·10
20	30·70	37	16·70
21	29·24	38	16·20
22	27·91	39	15·80
23	26·70	40	15·40
24	25·60	41	15·00
25	24·60	42	14·70
26	23·70	43	14·30
27	22·80	44	14·00
28	21·90	45	13·70
29	21·20	46	13·40
30	20·50	47	13·10
31	19·90	48	12·90
		49	12·60
		50	12·30

Hydrogen Ion Concentration

The determination of hydrogen ion concentration or pH of jam is an important operation to be carried out frequently and rapidly to ensure that the jam will set properly. Because the result is usually required quickly, it is most frequently obtained colorimetrically by the use of indicators, but it should be possible occasionally to check the result electrometrically.

FIG. 3.—PORTABLE pH METER.

This instrument is designed for measurements of pH on site. In addition to the meter itself a glass electrode system is mounted on the side of the box.

(*Cambridge Instrument Co., Ltd.*)

THE LOVIBOND COMPARATOR.—The simplest and most effective way of determining pH colorimetrically is by means of the Lovibond Comparator. This consists of a moulded case which opens out like a book. In the back portion are two cells or test tubes to hold the liquid under test, while in the front portion or lid is fitted a moulded disc which is fitted with nine glass colour standards. This disc can be rotated on the centre hub. Also in the lid are two holes through which can be observed the colours of the two cells containing the liquid. As the disc is rotated, each of the glass colour standards passes in turn in front of the left hand cell which is used as a blank and can be compared with the colour of the right hand cell. As the disc is rotated the value of the colour standard visible in the left hand

aperture appears at the indicator recess near the bottom right hand corner of the comparator case.

In measuring pH values of jam it is convenient to use the 20 per cent solution prepared for the invert sugar estimation as the two tests are usually made together. Both cells in the comparator are filled to the 10 ml. mark with the solution and to the right hand tube only is added 0·5 ml. of brom. phenol blue indication. With the appropriate colour disc in position the comparator case is closed. The disc is revolved until the nearest match is made and the pH value is read in the recess at the bottom right hand corner. Further details of the Lovibond comparator and the various types of pH meters will be found in *Chemical Engineering Instruments and Control Methods*.

FIG. 4.—A TYPE OF DIRECT-READING pH METER.

The Cambridge Instrument Co., Ltd.)

The above method has great advantages in simplicity and cheapness and it is reliable even in the hands of unskilled workers. The pH can of course be determined electrometrically by use of the standard type of pH meter using a glass electrode. Instruments are now available to give direct reading or an outfit can be fitted in the factory pipe line which will give a continuous indication or record of the jam as it is being made. This arrangement of course is only applicable to large installations.

Sulphur Dioxide (*see* also p. 177)

The amount of residual SO_2 in jam should be checked from time to time to ensure that it complies with legal standards. This applies particularly to strawberry jam where the fruit is very retentive of the preservative.

The apparatus used consists of a 1000 ml. round bottomed flask fitted with a two holed rubber stopper which carries a dropping funnel and a splash head connected at its other end to a water condenser. The water condenser which can be of the Leibeg type carries an adaptor for bubbling the distillate into water.

100 gm. of jam are weighed and washed into the 1000 ml. flask with distilled water, making the amount of distilled water used up to about 300 ml. The flask is connected to the remainder of the apparatus and it is arranged that the adaptor dips under the surface of about 300 ml. of water in a 500 ml. beaker. To this water is added a drop or two of 0·05 N. iodine solution and a few drops of starch solution. All distilled water used should be previously boiled.

By means of the dropping funnel about 20 ml. of concentrated hydrochloric acid is run into the flask which is then heated so that the boiling occurs in about two minutes. From a burette 0·05 N. iodine solution is run into the beaker as the distillation proceeds so that the blue colour of the "starch iodide" just remains. The end point is taken when the blue colour due to 0·1 ml. of iodine persists for one minute. The SO_2 content is calculated in parts per million from the formula

$$SO_2 \text{ in p.p.m.} = 8 \times T$$

where T is the titration in ml. with 0·05N. iodine solution.

Strength of Pectins, Lead Number and Colour

Other determinations which can usefully be made in the laboratory but which are not of a routine nature are the grade strength of pectins, lead number of jam and the colour of jam.

The strength of pectins is obtained by preparing a jelly under standard conditions and testing its strength by means of either the B.A.R. Jelly Tester[1] or the California Exchange Ridgelimeter.[2] For the determination of the approximate proportion of fruit present in jam a method has been described by Hinton[3] in which the quantity of precipitate formed when lead acetate is added to jam gives an indication of the amount of fruit in the sample. The colour of jam can be objectively assessed by means of the Lovibond-Schofield Tintometer. In this instrument standard coloured glasses are used to match the colour of the jam which is held in a square porcelain dish.

<div align="right">J. H. C.</div>

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CHAPTER 15

EDIBLE FATS

OILS and fats are complex mixtures of mixed triglycerides, (i.e., esters of fatty acids and glycerol). It may be necessary to examine them in several different forms, namely as crude oil, as refined oil after they have been subjected to refining processes, or as hydrogenated fats; as single fats or as blends of several components in margarine and shortenings.

THE PRODUCTION OF EDIBLE OILS AND FATS

The crude oils, obtained from vegetable seeds by expelling or extraction with solvents, and from animal tissues by rendering, will contain suspended impurities, moisture and free fatty acids and it is usual for the crude oil to be purchased upon a specification limiting these impurities. The production of edible oils usually involves alkali-refining, bleaching and deodorisation of crude oils. These processes are designed to remove from the crude oils the above impurities, together with substances responsible for colour and unpleasant, odoriferous materials such as those in the natural crude oils or those associated with rancidity. Further, deodorised oils which, when fresh, should be bland and odourless and of light colour, deteriorate if kept under unsuitable conditions or for a long time. Such deterioration results in the development of ill-flavoured oxidation products having the typical odour and flavour of rancidity.

Hydrogenation involves the catalytic addition of hydrogen to highly unsaturated liquid oils thereby converting them into solid fats. By this means, oils like fish and whale oil may be transformed into fats with characteristics suitable for edible purposes. Hydrogenation may be so controlled that by the choice of suitable hydrogenation conditions and by blending, it is possible to produce fats of almost any required texture and stability.

To test adequately an edible fat, whatever its form, it is necessary to:

1. determine the nature of the fat or fat blend to see whether it is of the type required or specified:
2. assess the quality of the fat.

In order to investigate these two factors completely would require a large number of tests ranging from simple physical determinations such as melting-points, to complex chemical analyses involving the use of expensive instruments. It is beyond the scope of this chapter to describe them all and many useful methods have, of necessity, been omitted. However, it is possible to outline a scheme which, when applied and interpreted in the light of experience should make possible the solving of most of the problems encountered in the day-to-day handling of edible fats. When describing techniques in detail, the methods of the British Standards Institute (B.S. 684) will be followed, if available, but where space will not permit detail, the methods will be discussed in general terms. Readers who wish to proceed beyond the limits of this chapter and have the necessary laboratory equipment available, are referred to the excellent standard text-books on the subject.

IDENTIFICATION OF FATS

The minimum amount of information required in order to attempt an identification of a fat or a mixture of fats is

(a) the mean molecular weight or chain-length of the constituent fatty acids,

(b) their mean unsaturation,

(c) the nature of the unsaturation, and

(d) the melting-point of the fat itself.

The first of these characteristics may be determined from the saponification equivalent or value and the second is measured by the iodine value; two tests which are the most useful and widely used in the analysis of fats. The nature of the unsaturation is revealed by estimating the possible unsaturated acids, linolenic, linoleic, oleic and iso-oleic acids. Linolenic, linoleic and oleic acids contain three, two and one double-bond, respectively, while iso-oleic acids are isomers of oleic acid and are indicative of the presence of hydrogenated fats.

Saponification Value and Equivalent

The saponification value of an oil or fat denotes the weight of potassium hydroxide expressed in milligrams, required to saponify one gram of the oil or fat.   The saponification value is related to the molecular weight of the fat and from it can be calculated the saponification equivalent, which is the amount of oil or fat saponified by 1 gram-equivalent of potassium hydroxide and is equal to 56,120 divided by the saponification value.

Weigh accurately about 2 gm. of the oil or fat into an alkali-resistant flask and boil continuously for one hour under a reflux condenser with 25 ml. (accurately measured) of alcoholic potassium hydroxide solution.   The contents of the flask must be swirled frequently.   (The alkali solution should not be less than 0·5 N., not darker than pale yellow, and prepared by dissolving potassium hydroxide in 95 per cent v/v ethyl alcohol; after standing for a few hours it is filtered.)   Determine the excess of alkali by titration with 0·5 N. hydrochloric acid using phenol phthalein, or methylene blue for dark solutions, as indicator.   The solution must still be hot when the end-point is reached.   A blank determination is made upon the same quantity of alkali at the same time and under the same conditions.

The acid solution must be accurately standardised, preferably using pure succinic acid under the above conditions.

Then if X = blank titration
          Y = sample titration
          W = weight of fat taken
          F = normality of acid solution

$$\text{Saponification Value} = \frac{(X\text{-}Y) \times 56\cdot12 \times F}{W}$$

Most common fats have saponification values between 190 and 200, with the exception of the "nut" oils, palm kernel and coconut oils, which are higher (240 to 250).   Thus high values denote the presence of the "nut" oils or butter fat, which may be distinguished by Reichert-Polenske-Kirschner values, a measure of the types of steam volatile fatty acids, which cannot be described in detail here.   Low values may denote the presence of some fish oils (e.g., cod oil), of rape-seed oil or possibly of mineral oil or other oils of high unsaponifiable content such as shark oil.   The saponification value of a mixture may be calculated from the saponification values of its components by direct proportion, but with saponification equivalents, their reciprocals must be used.

Unsaponifiable Matter

Unsaponifiable matter consists of that material present in oils and fats which is not saponifiable by caustic alkali. It can be used to detect the presence of mineral oils and the higher alcohols such as cholesterol and phytosterol, which in the form of their acetates may be used to distinguish between animal and vegetable fats.

Saponify approximately 2 gm. of the oil or fat, accurately weighed, as for the saponification value. Transfer the contents of the flask to a 250 ml. separating funnel, washing it with 50 ml. of water. Rinse the flask with 50 ml. of ethyl ether and pour into the funnel. Shake the funnel vigorously while the contents are still warm and allow the funnel to stand until the two layers of liquid separate. Draw off the aqueous layer into the saponification flask.

Pour the ethereal solution into a second funnel containing 20 ml. of water. Extract the soap solution twice more with 50 ml. of ether and combine the three extracts in the second funnel. Rotate the extracts gently with the 20 ml. of water and after allowing to separate, run-off the wash water. Wash the ethereal solution twice with 20 ml. of water, shaking vigorously on each occasion. Successively wash with 20 ml. of 0·5 N. aqueous potassium hydroxide and then 20 ml. of water, three times. Continue washing with water until the water wash no longer turns pink on addition of phenol phthalein.

Transfer the ethereal solution to a weighed flask and evaporate to small bulk. Add 2 to 3 ml. of acetone and completely remove the solvent by means of a gentle current of air, the flask being rotated in boiling water or on a steam bath. Dry the flask and contents to constant weight at a temperature not exceeding 80° C.

Iodine Value

The iodine value of an oil or fat is expressed as the percentage of iodine absorbed under the conditions of the test and is a measure of the unsaturation of the fat.

Weigh accurately a convenient quantity of the oil (0·1 gm. for iodine values of 180; 0·2 gm. for 100 and 1 to 2 gm. for 20) and dissolve in 10 mls. of carbon tetrachloride in a clean, dry glass bottle of about 500 ml. capacity, provided with a well-fitting glass stopper. Add 20 ml. of Wijs solution (prepared as described below) and seal the bottle by moistening the stopper with a 10 per cent solution of potassium iodide.

Allow the bottle to stand for 30 minutes (1 hour for linseed oil) in a dark place. Add 15 ml. of a 10 per cent solution of potassium iodide and 100 ml. of distilled water and titrate the free iodine with standardised sodium thiosulphate (approximately 0·1 N.), adding starch as indicator as near the end-point as possible. Make a blank determination upon the same quantity of reagents, at the same time and under the same conditions.

Then if X = blank titration
         Y = sample titration
       W = weight of fat taken
        I = weight in gm. of iodine equivalent to 1 ml. of the
           thiosulphate solution

$$\text{Iodine Value} = \frac{(X-Y) \times I \times 100}{W}$$

PREPARATION OF WIJS SOLUTION

Dissolve 10 gm. of iodine trichloride in 300 ml. of carbon tetra-chloride and 700 ml. of glacial acetic acid. Titrate 25 ml. of this solution with 0·1 N. thiosulphate solution as above, without standing. The titration should be 34 to 37 ml. and if not must be suitably adjusted.

Then if V = volume in ml. of whole of iodine trichloride solution
        A = titration
the iodine equivalent in grams of whole solution is:

$$\frac{V \times A \times I}{25}$$

To the whole solution add 0·55 times the iodine equivalent of pure iodine and dissolve. Do not use for three days after preparation.

Thiocyanogen Value

The thiocyanogen value of a fat denotes the percentage by weight of iodine calculated from the thiocyanogen absorbed under the condition of the test. Whereas thiocyanogen, like iodine, unites quantitatively with oleic acid or other monoethenoid groups present, it combines with only approximately one double bond (53 per cent) in linoleic acid and two (60 per cent) in linolenic acid. Consequently, for fats containing these acids, the determination of both iodine and thiocyanogen values affords a method for estimating the three unsaturated acids.

All reagents and apparatus must be completely dry, the latter being dried immediately before use at about 120° C. for at least one hour.

Weigh 0·1 to 0·2 gm. of the fat into a 50 ml. stoppered bottle. If the fat is solid add 5 ml. of anhydrous carbon tetrachloride to ensure solution. Run 25 ml. of the thiocyanogen reagent (prepared as below), with a minimum of exposure to air into the bottle, shake well and then place in the dark for 15 to 16 hours at 15° C. to 20° C. 20 ml. of saturated potassium iodide are then added, followed by about 200 ml. of water and the liberated iodine titrated in the usual way with 0·1 N. thiosulphate solution. A blank determination is carried out at the same time.

Then, as for the iodine value, the thiocyanogen value is:

$$\frac{(X-Y) \times I \times 100}{W}$$

The 0·1 N. solution of thiocyanogen is prepared as follows:

Pour 500 ml. of cold anhydrous glacial acetic acid into each of two perfectly dry, stoppered bottles. Add 8·4 gm. bromine to one bottle and 25·7 gm. lead thiocyanate to the other. Add the solution of bromine in acetic acid to the suspension of lead thiocyanate with shaking and continue the shaking for 2 or 3 minutes; then, intermittently every 10 minutes until the solution is colourless; the whole operation usually taking 30 minutes. Filter the thiocyanogen solution rapidly through a dry filtration apparatus. The clear solution, which must be free from any pink coloration should be kept in an amber glass-stoppered bottle in the dark at 15° C. to 20° C. and used within three days.

Fatty acid compositions may be calculated from the following iodine and thiocyanogen values:

Fatty Acid	Iodine Value (J)	Thiocyanogen Value (T)
Oleic	89·9	89·4
Linoleic	181·1	93·9
Linolenic	273·5	162·5

When no linolenic acid is present.

Percentage of oleic acid (X) $= 2\cdot339T - 1\cdot214J$
Percentage of linoleic acid (Y) $= 1\cdot155J - 1\cdot160T$
Percentage of saturated acids $= 100 - X - Y$

If linolenic acid is present, then the percentage of saturated acids (S) must be determined in some other way.

Percentage of oleic acid $= 1\cdot5829T - 1\cdot1751J - 0\cdot6416S + 64.16$
Percentage of linoleic acid $= 1\cdot2531J - 3\cdot1466T - 1\cdot6855S + 168\cdot55$
Percentage of linolenic acid $= 1\cdot5637T - 0\cdot0780J + 1\cdot3271S - 132\cdot71$.

As traces of moisture can seriously affect the accuracy of the determination it is always as well to carry out the tests in duplicate.

A spectrophotometric method is the most convenient and most accurate way of determining linoleic and linolenic acids, if a spectrophotometer is available.

The method consists of partially conjugating the acids with an alkali solution under carefully standardised conditions and measuring their absorption of ultra-violet light. Details of the method, however, must be sought elsewhere.

Solid Acids and Iso-oleic Acids

Prepare fatty acids from the fat by saponifying completely with alcoholic potash, and splitting with dilute sulphuric acid. Extract the fatty acids with ether, wash the ethereal solution free from acid and dry it with sodium sulphate. Distil off the ether, removing the last traces under vacuum.

Dissolve 3·5 gm. of freshly prepared fatty acids and 3·45 gm. of lead acetate in two separate 50 ml. portions of 92 to 93 per cent alcohol. (With liquid oils or fats containing less than 25 per cent of solid acids only 1 gm. of lead acetate is used.) Heat both solutions to boiling and pour that containing the lead acetate into the one containing the fatty acids. Mix thoroughly, heat again to boiling-point and allow to cool slowly. The solution is allowed to stand overnight at 15° C. to 20° C., which temperature must be strictly adhered to by means of a maximum-minimum thermometer. Stir the mixture and filter on a 10 cm. Buchner funnel which is then transferred to a clean filter flask.

Wash the lead salts in the funnel and any remaining in the filter flask with 100 ml. petroleum ether (40° C. to 60° C. boiling-point) and distil off the petroleum ether completely on a water-bath. Dissolve the residue by boiling under a reflux condenser with 20 ml. 92 to 93 per cent alcohol containing one drop of glacial acetic acid. Crystallise this solution for 3 hours at 15° C. to 20° C. Filter off the precipitated lead salts and wash with 20 ml. of cold 92 to 93 per cent alcohol. Transfer the lead salts from both funnels to a beaker and decompose them with dilute nitric acid and recover the acids with ether. The total solid acids are weighed and the whole or portion used for an iodine value determination.

$$\text{Percentage of iso-oleic acid} = \text{Percentage of total solid acids} \times \frac{Iodine\ Value}{90}$$

Melting-Points

Fats are such complex mixtures of glycerides which melt at different temperatures, that no sharp melting-point can be measured. However, a comparable, approximate melting-range of a fat may be determined by the following methods.

Introduce about 1 cm. length of the fat into a closed capillary tube of 3 mm. bore and keep in a refrigerator overnight or for at least 3 hours at 0° C. Attach the tube to a thermometer using rubber bands so that the end of the tube is level with the thermometer bulb. Suspend the thermometer in a beaker, 3 cm. below the surface of the water and slowly heat at a rate of about 0·5° C. per minute with continued stirring. When a clearly defined meniscus is observed record the temperature as the Incipient Fusion. Continue heating until the fat is just free from unmelted particles and record the temperature as the Complete Fusion.

o

An Open-Tube melting-point, or slip-point, is probably the most reliable single melting-point of a fat.

Dip a capillary tube of 1 mm. bore and open at both ends, into the fat until a column of fat 1 cm. in length is formed. Chill and heat as above and record the temperature at which the fat commences to rise in the tube; record this as the Open-Tube melting-point.

If a single figure is required the Open-Tube melting-point is most characteristic but all three may be declared as a measure of the melting-range of the fat. A more complete picture of the melting characteristics of a fat may be obtained by means of dilatometric measurements which are too lengthy for inclusion here.

Other physical characteristics such as specific gravity, viscosity and, particularly, refractive index are of use to the oil analyst and are determined by standard procedures which will be familiar. The refractive index is governed chiefly by the proportion and degree of unsaturation and is of particular value in following the course of hydrogenation.

The tables opposite give the values which would be expected for typical samples of the common edible fats when tested by the methods which have been described.

ASSESSMENT OF THE QUALITY OF EDIBLE OILS

It may be necessary to assess the quality of either "crude" oils or "refined and deodorised" oils. For the former the specified criteria are usually moisture, impurities, colour and free fatty acid content, while for the latter, flavour, free fatty acid content, colour, rancidity and stability to autoxidation are the minimum requirements for reliable judgement.

Moisture

Several methods are available for estimating the moisture content of an oil including methods by which the volatile matter is driven off at elevated temperatures, the Dean & Stark method which utilises distillation with an immiscible solvent (toluene or heptane) and the Karl Fischer method. The simplest and quickest method for estimating volatile matter, which in most, though not all, cases is moisture, is as follows:

TABLE 1

Oil or Fat	Sap. Value	Sap. Equiv.	Per Cent Unsap.	Iodine Value	Per Cent Linoleic Acid	Open Tube M.pt. °C.	Refractive Index
Coconut Oil .	255–260	215–220	0.2	7–10	1·5	24–28	1·449/40° C.
Palm Kernel Oil	243–250	225–230	0·2–0·5	14–19	1·0	25–30	1·450/40° C.
Cacao Butter .	192–195	286–292	0·3–0·8	35–42	2·0	33–35	1·450/60° C.
Palm Oil .	196–210	256–285	0·3–0·6	51–58	10·5	30–42	1·451/60° C.
Olive Oil .	188–195	287–298	0·5–1·5	79–88	7·5	—	1·470/20° C.
Groundnut Oil	188–196	286–298	0·3–0·8	82–99	25·0	—	1·470/20° C.
Cottonseed Oil.	192–196	286–292	0·7–1·6	103–113	48·0	—	1·473/15° C.
Sesame Oil .	188–193	291–298	1·0–1·5	105–118	40·5	—	1·474/20° C.
Sunflower-seed Oil	186–194	290–300	0·3–0·5	120–140	57·0	—	1·473/25° C.
Soyabean Oil .	190–193	291–296	0·6–1·2	129–143	51·0(8·5% linolenic)	—	1·475/20° C.
Butter Fat .	216–235	237–260	0·3–0·5	26–45	3–4	28–33	1·460/25° C.
Lard . .	193–200	280–290	0·2–0·4	46–66	6·0	28–48	1·441/60° C.
Beef Tallow .	190–200	280–295	0·2–1·0	32–47	2–3	40–50	1·451/60° C.
Mutton Tallow .	192–198	283–291	0·2–1·0	31–47	3–4	44–49	1·450/60° C.

Weigh accurately into a tared dish 8 to 9 cm. dia. and 4 to 5 cm. deep, preferably flat-bottomed, 10 to 15 gm. of the prepared sample. Heat in an oven at 105° C. for 1 hour. Cool in a desiccator and re-weigh. Re-heat at the stipulated temperature for a further half-hour. Cool and re-weigh. Continue to re-heat, cool, and re-weigh until the loss of weight between consecutive weighings does not exceed 1 mg. Express the loss of weight as the percentage of volatile matter.

Impurities

The impurities or total dirt in an oil or fat consist of the sum of all mineral matter present together with the organic "dirt" or organic constituents of those substances in the oil, exclusive of water and volatile matter, which are not dissolved by a specified solvent applied under specified conditions.

(a) TOTAL DIRT.—Filter a quantity of from 20 to 50 gm. of the oil or fat at a temperature below 60° C. through a filter paper previously dried in the oven at 105° C. and weighed in a stoppered weighing-bottle. If the oil or fat is slow in filtering it may be diluted prior to filtration with light petroleum (b.pt. 40° C. to 60° C.). Extract the filter paper containing the impurities with light petroleum (b.pt. 40° C. to 60° C.) in a continuous extraction apparatus. After complete extraction, dry the filter paper and contents in an oven at 98° C. to 100° C. till constant in weight. Calculate the percentage of total dirt.

(b) ORGANIC DIRT.—Ash the filter paper and residue from (a) above in a suitable crucible. Deduct from the weight of ash found the weight of the ash of the filter paper. Deduct the difference from the weight of total dirt (see (a) above). Calculate the percentage of organic dirt.

Colour Reading

The colour of an oil is of considerable importance commercially and consequently an agreed standard of comparison is necessary. The colours of oils are thus usually declared in Lovibond units, and may be measured in an instrument designed for the purpose.

Such an instrument includes a standard light source, glass-ended cells of accurate length (1 in. and $5\frac{1}{4}$ in.) and the series of coloured glasses which form the basis of the system. The important property of these glasses is that they are additive. There are three series, yellow, red and blue and the colour of a given oil may be matched by a suitable combination of them. It is usual to avoid using blue although this is not always possible, and it is best to keep the number of matching glasses to a minimum. The oil must be clear and bright and to achieve this may be filtered through a filter paper if necessary. It is important to avoid excessive heat which may affect the colour of oils.

It is, of course, essential for success in colour matching that the

operator should be free from colour-blindness and too many colours should not be read at one time as tiredness affects the judgement of the eye.

Free Fatty Acids

Weigh into a flask a suitable quantity of the oil or fat according to the colour and degree of acidity—2 to 50 gm. (the titration should not exceed 10 ml.). In a second flask bring some alcohol to the boiling-point and while still above 70° C. neutralise it with 0·1 N. aqueous alkali using 0.5 ml. of phenol phthalein as indicator. Pour 50 ml. of the neutralised alcohol onto the oil in the first flask and mix the contents. Bring them to the boil and while as hot as possible titrate with 0·1 N. or 0·5 N. aqueous alkali solution shaking vigorously during the titration. The end-point of the titration is reached when the addition of a single drop produces a slight but definite colour change which persists for at least 15 seconds.

The acidity may be calculated as the acid value for all fats or as the percentage of free fatty acid based upon the molecular weights of:

Lauric acid (200) in the "nut" oils (palm kernel and coconut oils).
Palmitic acid (256) in palm oils.
Oleic acid (282) in all other oils.

The acid value is defined as the number of milligrams of potassium hydroxide required to neutralise the acidity in 1 gm. of the oil or fat.

Then if X = titration
        W = weight of oil taken

$$\text{the acid value} = \frac{X \times 5\cdot61}{W}$$

and the percentage of free fatty acids =

$$\frac{X \times \text{molecular weight}}{W \times 100}$$

The percentage of free fatty acids in crude oils varies considerably but in deodorised oils should be no more than 0·10 per cent.

Organoleptic Tests

The simplest and most obvious tests for deterioration in fats are based upon taste and smell. Correlation of chemical data with flavour has, as yet, been very uncertain and indecisive. Consequently, as flavour is the main criterion by which an edible oil is judged, panels of tasters are frequently the final arbiters of quality. A single person, with experience, may be sufficiently reliable to exercise adequate routine control, but, as various psychological factors may be involved, it is well to have a panel of four.

By giving a numerical value to a fat which is completely bland and odourless and a second arbitrary number to a fat, the flavour of which is just unacceptable, an agreed scale may be established with practice, in which equal increments correspond to equal increments of flavour intensity. By these means it is possible to classify fats accurately, not only according to the amount of flavour but also according to its nature. Where decisions rest upon the tastes of individuals, with all their idiosyncrasies, it is necessary to keep a careful check upon results and to treat them statistically wherever possible.

Peroxide Value

Off-flavours which develop in fats are usually caused by autoxidation which eventually results in the objectionable taste and odour recognised as rancidity. The initial oxidation products which are formed are hydroperoxides and these are usually measured by iodimetric methods. The simplest and most useful for routine purposes is that of Lea.

Weigh 1 gm. of the oil or fat into a pyrex test-tube (6 in. × 1 in.) which must be thoroughly clean. Add approximately 1 gm. of powdered potassium iodide and 20 ml. of glacial acetic acid—carbon tetrachloride (or chloroform) mixture (2:1 by volume) and heat the liquid to boiling-point over a small flame impinging on the bottom of the tube. Continue the boiling in a beaker of boiling water for 30 seconds, the heavy vapour of the liquid minimising the diffusion of air back into the tube. Cool the tube rapidly under the tap, pour the contents into 30 ml. of water and titrate with 0·002 N. sodium thiosulphate solution using freshly prepared starch solution as indicator. A blank test should be carried out upon the reagents but should be negligible.

The peroxide value is expressed as ml. of 0·002 N. sodium thiosulphate solution per gram of sample. The peroxide value of a freshly deodorised fat should be nil and a fat received directly from the processor should seldom exceed a peroxide value of 1·0. Most fats are deemed rancid when their peroxide values are between 10 and 20.

Rancid flavours, however, are not due to the peroxide themselves but to their decomposition products, which are carbonyl compounds. Thus it is possible to have an oil which has a low peroxide value and yet is very rancid, the pre-formed peroxides having broken down into their ill-flavoured decomposition products. Great care should therefore be exercised in judging

the state of oxidation of an oil on peroxide value alone, if the previous history of the oil is not known.

Two methods have been extensively used to assess rancidity due to these secondary oxidation products and will now be described.

Kreis Test

Shake vigorously 10 ml. of the oil or melted fat for 30 seconds with 10 ml. of concentrated hydrochloric acid (sp. gr. 1·19). Add 10 ml. of a 0·1 per cent solution of phloroglucinol in ether and shake the mixture again. If a red colour develops, dilute the sample with a non-reacting substance (such as kerosene) until no such colour develops. A positive test for a dilution of 1 part of oil in 20 parts of kerosene would indicate a degree of rancidity evident to taste and smell. Care must be taken not to interprete this test too rigidly as it is possible to get a red colour with an oil which is not rancid.

Issoglio Test

Heat 25 gm. of the oil or fat with 100 ml. of distilled water on a steam bath for two hours with constant shaking. Separate the aqueous portion from the mixture by passing it through a wet filter paper and make up the filtrate to 100 ml. with distilled water. Boil 10 ml. of the filtrate for 5 minutes with 50 ml. of 0·01 N. potassium permanganate solution; cool the contents of the flask and add 10 ml. of sulphuric acid (1 in 5) and 50 ml. of 0·01 N. oxalic acid solution. Heat the solution and titrate with 0·01 N. permanganate. If T and t represent the volumes of permanganate solution used up in the oxidation and in a blank test respectively, and W the weight of the fat taken, then the "oxidisability value," which represents the number of milligrams of oxygen required to oxidise the water-soluble constituents from 100 gm. fat, is given by:

$$\frac{(T - t)\ 80}{W}$$

Fresh fats give values from 3 to 10, while a figure of over 15 suggests excessive rancidity.

Neither of these tests can be relied upon to correlate directly with rancidity and high values should be considered only as indicative of deterioration, implying the need for further examination. Recently, various colorimetric methods have been published for the estimation of carbonyl compounds in very small concentrations and it is possible that one such method may prove more successful and useful.

INDUCTION PERIODS

The above tests give some indication as to the state of oxidation of an oil at the time of the test but cannot be used to indicate how long a given oil will keep before becoming rancid. Accelerated tests have therefore been devised which are reproducible, which can be correlated to "shelf-life" under normal conditions and with which an answer can usually be obtained within the limits of the working day. These methods include storage at suitable elevated temperatures; oxygen absorption methods using such apparatus as the Barcroft-Warburg absorptiometer with which the rate of oxygen absorption may be measured at selected temperatures; and aeration methods. As it is possible to describe only one, a modification of an aeration method known as the Swift Active Oxygen Method has been selected, as the Swift test is used extensively in the United States and requires no special apparatus.

The Swift Active Oxygen Method

20 ml. of fat are measured into a test-tube (8 in. × 1 in.), which is placed in a bath maintained at 98° C. Clean, dry air is passed through the fat at a steady rate and the peroxide value measured at intervals depending upon the predicted stability of the fat. The rate of oxidation may be followed closely by plotting a graph of the peroxide value against time. With vegetable oils the increase in peroxide value is slow at first but at the induction period a rapid acceleration takes place which is clearly discernible on the graph which turns sharply upwards. For many fats this is in the neighbourhood of peroxide value 20 and frequently the number of hours required to produce a peroxide value of 20 is termed the induction period.

The air may be cleaned by bubbling it through chromic acid and then water and finally drying it with a calcium chloride tower. From the tower it may be passed into a large jar with several delivery tubes so that a number of tests may be run at the same time from the same "purifying train." It is essential that the aeration tubes should be kept scrupulously clean as contamination invariably affects the induction period. One advantage of the method is that the air leaving the aeration tube may be constantly inspected for rancid odours. Although there are no accepted standards the above test may be used successfully for the routine comparison of the oxidative stability of oils.

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A NOTE ON BEEF FATS

PREMIER JUS.—This is the fat of the heart, caul and kidneys, rendered at 100° F. to 120° F.

BEEF STEARINE.—The harder portion which remains when the more liquid oily fraction of premier jus is removed by hydraulic pressure.

OLEO OIL.—The expressed oil referred to above.

TALLOWS.—These are of various grades and are derived from the rendering of the fatty portions of the carcase not used for premier jus.

DRIPPING.—"Dripping" means unbleached, unadulterated fat:

(a) produced from, or by the rendering or processing of, the fat or bones of sheep or oxen;

(b) of a sweet taste;

(c) of a sweet smell;

(d) untreated by any chemical process; and

(e) containing not less than 99 per cent saponifiable matter and not more than 1·5 per cent free fatty acids; but does not include imported premier jus.

Some typical analytical figures for these products are:

	BEEF TALLOW	PREMIER JUS	BEEF STEARINE	OLEO OIL
Melting-point °C. .	47–49·5	47–49	50–54	Variable
Saponification value	193–199	195–200	192–197	198–202
Iodine value . .	38–44	38–45	18–25	40–51
Acid value as oleic per cent . .	0·2–1·5	0·2–0·5	0·1–0·3	0·25–0·6

Sampling

Melt fat, and filter by means of a hot water funnel. Make the different determinations on samples of this melted homogeneous mass.

Weigh out at one time as many portions as are needed. Keep fat in a cool place and protected from light and air, otherwise it will soon become rancid.

The usual analyses and tests carried out (by methods already described) are: slip point, iodine value, acid value, saponification value and rancidity.

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CHAPTER 16

WHEAT TESTING

1. Moisture Content

A FIGURE for the moisture content of wheat can be obtained in various ways but the magnitude of the figure is dependent upon the method employed, being influenced in drying methods, for example, by the temperature and duration of the heating operation.

A procedure widely used in Britain is to heat 5 gm. of a coarsely ground sample of wheat for 5 hours at 100° C., although it may be more convenient in some circumstances to use 10 gm. and to dry at 100° C. overnight. Quicker methods involving the use of specially designed drying ovens are available; in one the wheat is heated for 1 hour at 130° C. and in another for 15 minutes at 155° C. Rapid electrical methods are also available but are usually less accurate when the moisture content of the wheat is high.

A hand-operated coffee mill is suitable for grinding the wheat prior to determining its moisture content but the heat engendered during the grinding may cause some loss of moisture if the wheat has a moisture content of, say, 18 per cent or more. Such figures are encountered in native wheat in some seasons. In such circumstances, a sample of the wheat should be partially dried in the unground state, being weighed before and after the drying, and the grinding and completion of the drying then performed.

The moisture content of wheat varies with the variety and within any one variety is likely to vary from season to season. Australian wheat is usually dry, with a moisture content in the region of 10·5 per cent, while the figure for Manitoban is often of the order of 13 per cent. Wheat grown in Britain gives much higher figures; moisture contents of 16 per cent are common and figures of 20 per cent not infrequent. The miller must know the moisture content of each wheat before he uses it because the amount of moisture naturally present will partially determine the amount which he will add during the conditioning process.

2. Protein Content

The protein content of wheat is calculated from the nitrogen content, which is determined by the Kjeldahl procedure (*see also* p. 225).

The wheat sample should be coarsely ground on a hand-operated coffee mill and a 1 gm. aliquot of the ground material used for the test. Selenium is a suitable catalyst.   In the author's laboratory 1 gm. of the ground wheat, 20 ml. of concentrated sulphuric acid, 9 gm. of potassium sulphate and 0·05 to 0·1 gm. of powdered selenium are digested in a Kjeldahl flask for about an hour and, after being cooled and diluted, the liquid is made alkaline and distilled for about 40 minutes into 100 ml. of a 1 per cent solution of boric acid which has been neutralised to the indicator used in the subsequent titration (2 gm. water-soluble methyl red and 0·5 gm. methylene blue, dissolved in 500 ml. water). When the distillation has been completed, the distillate is titrated, using the above-mentioned indicator, to a grey-brown colour with N/14 hydrochloric acid.

The protein content of a flour will be lower than that of the wheat from which it is milled, the difference being about 0·5 to 1 per cent depending upon the extraction.   Suitable protein contents are 12 per cent in bread flour, 10 per cent in confectionery flour, and 8 per cent in biscuit flours.

The protein contents normally encountered in different types of wheat are given in Table I.

TABLE I

RANGES OF PROTEIN CONTENT FOUND IN VARIOUS WHEATS

	per cent
Manitoban . . .	11·3–14·3
Garnet . . .	9·9–11·4
Canadian Durum. .	10·5–12·9
Hard Winter . .	10·5–13·0
Russian . . .	10·3–13·7
Plate	10·3–14·2
Australian . . .	8·6–12·9
Danube Basin Wheats .	9·1–11·4
Soft White Pacific .	9·1–10·8
English, Irish and Continental . .	7·4–11·4

3.   Diastatic Activity—Maltose Figure

The diastatic power of a material is its ability to convert starch into sugar, and a standard method of measuring it is to extract the responsible diastatic enzymes, allow them to act under standardised and controlled conditions on a specially prepared soluble starch, and then determine the amount of sugar formed.   This method, however, measures only the action of the diastatic enzymes of the material on a fully susceptible starch, whereas in a bread dough the diastatic enzymes have to produce sugar by acting on the natural starch of the flour.   Only a portion of this starch is readily susceptible to diastatic attack and moreover the proportion of susceptible starch is not constant for all flours.   The application of the standard procedure—the Lintner test—to flours does not provide a reliable measure of their relative powers of producing sugar from their own starches. In order to obtain this information, it is necessary to use a procedure in which the starch of the flour under examination is the substrate upon which its diastatic enzymes act during the test. Such a procedure is available, and has been in use for 25 years as a means of measuring the diastatic activy of commercial flours.

Special considerations arise, however, when the diastatic activity of a wheat is under consideration.   In order to apply the procedure just mentioned, it is necessary to mill the wheat into flour, and the diastatic activity of the flour produced will vary with the milling procedure.   The explanation is that during the milling process some of the starch is damaged and thereby rendered susceptible to diastatic action.   The diastatic power of the flour produced from a given wheat will, therefore, be related to the degree of starch damage caused during the milling. Hence, the first criterion that has to be met when wheats are being tested for diastatic activity is that the milling of them into flour must be performed under controlled conditions which permit the amount of starch damage to be kept reasonably constant.   The fact that the extent to which the starch is damaged during the laboratory milling may not be the same as that which occurs during a commercial milling of the same wheat is of no moment because the difference can be adjusted by means of an experimentally determined factor.

Several types of laboratory mill are available for producing flour from relatively small samples of wheat.   The most reliable

are those which follow the basic principles of large scale milling, that is, employ several break rolls, several reduction rolls, and intermediate sieving operations which simulate the scalping, grading and flour dressing in a large mill.   With a model mill of this type it is possible to produce flour under such standard conditions that the diastatic activities of wheat can be determined without fear that the results are subject to a significant error due to differences in degrees of starch damage.

The method by which the diastatic activity of a flour produced from a wheat on a laboratory model mill is measured involves incubating a suspension of that flour for a given time at a given temperature and then determining the amount of sugar in the suspension.   The test is an empirical one in that the temperature and duration of the incubation have been arbitrarily chosen, but the results provided by the method have been well checked against gas production and baking tests and their significance is fully understood.   The procedure, which is known as the "maltose test," is as follows:

Weigh 15 gm. of flour into a clean dry 8 oz. bottle and place the bottle in a water bath maintained at 27° C. for about 15 minutes. Then add 95 ml. of water at 27° C., close the bottle with a rubber bung and shake thoroughly until a uniform suspension is obtained.

Remove the bung and place the bottle in the water bath.   Leave for exactly 1 hour, shaking the contents of the bottle gently at intervals of 15 minutes, and then add 1·5 ml. of dilute sulphuric acid (200 ml. concentrated sulphuric acid diluted to 1 litre) and 3·5 ml. of a 15 per cent solution of sodium tungstate.   Mix, filter through a No. 5 Whatman paper, and titrate the filtrate against a boiling mixture of 5 ml. of Fehling's solution A (69·28 gm. $CuSO_4. 5H_2O$ per litre) and 6 ml. of Fehling's solution B (350 gm. of sodium potassium tartrate and 100 gm. NaOH per litre) using methylene blue (1 per cent aqueous solution) as indicator.

The "maltose figure" of the flour, i.e., a measure of its diastatic activity, can be obtained from the titration reading by reference to Table II.

If the maltose figure of a bread grist is below 1·5 the possibility exists that under some bakery conditions a dough made from flour milled from the blend will fail to produce sufficient supplementary sugar by diastatic action to enable the yeast to continue producing gas throughout the fermentation period.   Satisfactory bread may be made from flour with a maltose figure of only 1·5

TABLE II

TABLE FOR CONVERSION OF COPPER REDUCING POWERS OF FLOUR EXTRACTS INTO "MALTOSE FIGURES"

ml. Flour Extract used in Titration	Maltose Figure
15	3·6
16	3·4
17	3·2
18	3·0
19	2·8
20	2·7
21	2·6
22	2·4
23	2·3
24	2·2
25	2·1
27	2·0
30	1·8
34	1·6
39	1·4
46	1·2
50	1·1

but there is little margin of safety, and a higher figure, even if artificially produced by the addition of malt flour, is desirable.

If the maltose figure of a blend is above 2·5, indicating that the sugar-forming enzymes are very active, there is danger that the allied dextrin-forming enzymes will also be active, in which event bread produced from flour milled from the wheat in question may have a sticky crumb.

4. Protein Quality

A knowledge of the physical properties of the protein in a wheat is essential if that wheat is to be used to the best advantage by the miller. The strength of the protein will determine whether a wheat proposed as a constituent of a bread grist is one which will carry weaker wheats, is one which ranks as a filler, i.e., can give no support but requires none itself, or is one which must be carried by strong wheats. The elasticity and extensibility

of the protein will reveal whether the wheat needs to be blended with wheats of a compensatory nature in order to counteract an excess of toughness or of flowiness. The strength, elasticity and extensibility of the protein of weak wheats will determine whether they are suitable for the production of biscuit flour.

Before the nature of the protein in a wheat can be reliably assessed, flour must be produced from the wheat so that a dough can be made. Moreover, the conversion of the wheat into flour must be performed under standardised and controlled conditions which are reproducible, so that differences found in the ultimate tests cannot be attributable to variations in the milling procedure. Reliable wheat testing, therefore, calls for the use of a model mill of the type recommended in the section on "Diastatic Activity." Each wheat should be suitably "conditioned," i.e., brought to the correct physical state for milling by being allowed to stand over night after being damped with the requisite amount of water, and then milled at fixed roll settings until a given extraction has been achieved.

DOUGH TESTING INSTRUMENTS

The protein characteristics of the flour obtained from a wheat can be objectively studied by means of mechanical dough testing instruments.

If such an instrument is to provide reliable and helpful information it is necessary that:

1. It should be sensitive, so that reasonably fine differences in dough properties can be detected.
2. The results it furnishes should be reproducible.
3. It should permit various physical properties of the dough to be evaluated separately.
4. It should provide assessments which can be expressed on a numerical basis.

Several dough testing instruments which meet these criteria are available and the principle upon which they operate is the extension of a piece of dough until it breaks, the operation being performed in such a way that the magnitude of the applied force and the degree of extension are automatically recorded throughout the test. In some of them, such as the Extensograph and the Extensometer, the stretching of the dough is performed by a

moving arm, whilst others, of which the Alveograph is the best known example, rely upon air pressure.

The Alveograph (Fig. 1) is a particularly useful instrument for assessing the protein quality and blending potentialities of wheats and it is much used for this purpose. It enables a
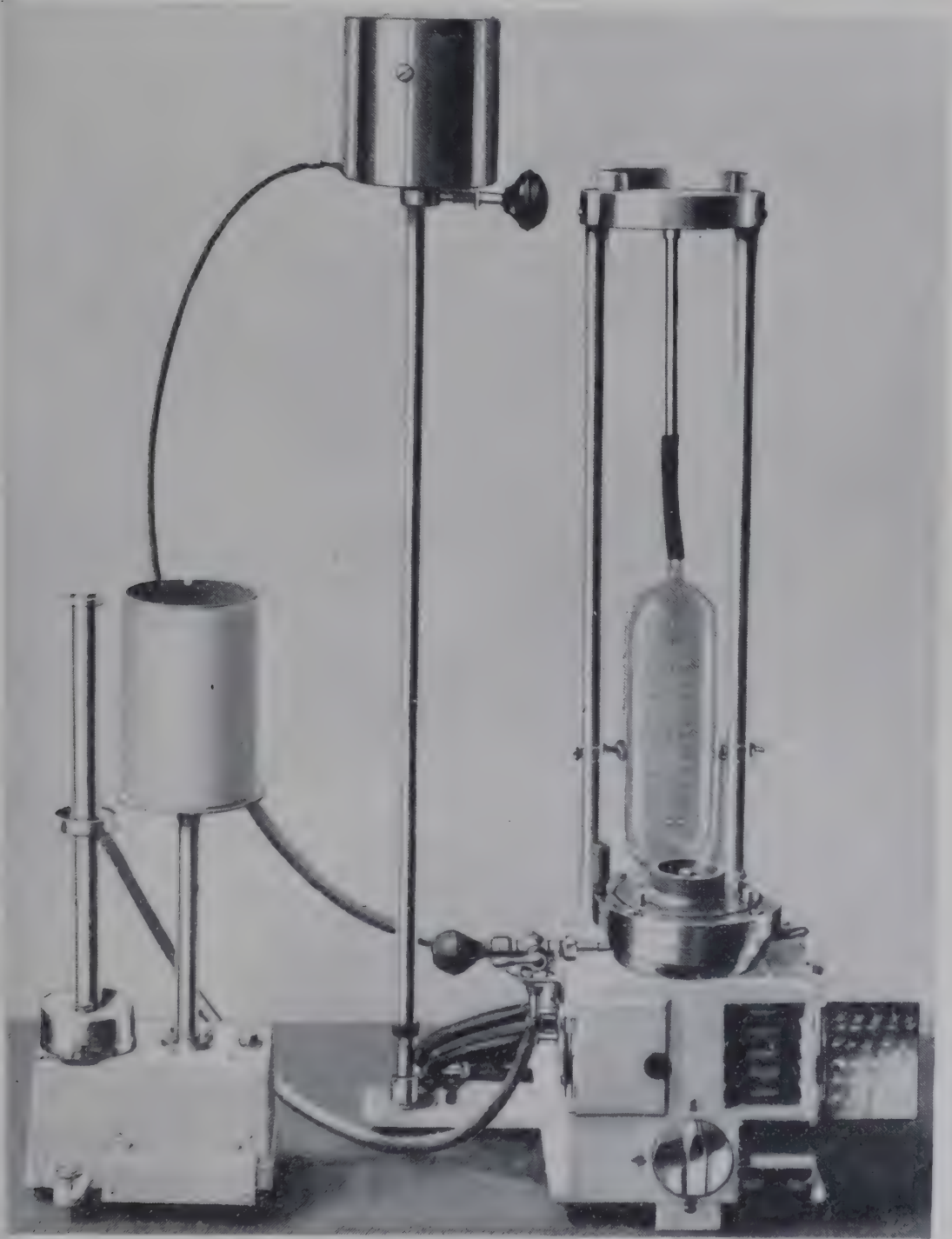
FIG. 1.—THE ALVEOGRAPH.

(*Thos. Robinson and Son, Ltd.*)

P

standardised disc of dough to be blown into a bubble in such a manner that the pressure within the bubble and the degree of extension of the dough from the time inflation commences until bursting occurs are continuously and automatically recorded. The dough is prepared in a specially designed mixer at constant temperature and is subsequently moulded by a standardised procedure. The doughs are made to a fixed water content, allowance being made for the moisture content of the flour.

A typical curve furnished by the Alveograph is depicted in Fig. 2. The measurements normally made on these curves are the height of the peak, "P," which is a measure of the stability of the dough; the length of the base line, "L," which is a measure of the extensibility of the dough; and the area enclosed by the curve, "W," which is an index of the strength of the dough. The relative magnitudes of these separate protein properties, i.e., the balance between them, is a most important factor, because lack of balance in a commercial flour will lead to criticism no matter how satisfactory is the strength of the flour. The shape of an Alveographe curve is, therefore, as important as its area.

The importance of the balance between the individual protein characteristics is illustrated in Fig. 3. The three curves "A," "B," and "C" are equal in area and hence represent flours of identical strength. Curve "A" is well-proportioned and represents a flour which would handle well in the dough. The flour responsible for curve "B" is identical in strength with flour "A" but is quite unsuitable for
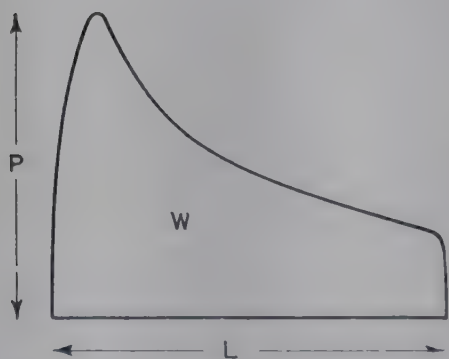
FIG. 2.—TYPICAL ALVEOGRAPH CURVE.

FIG. 3.—THREE ALVEOGRAPH CURVES OF EQUAL AREA.

breadmaking because it gives an excessively stiff dough lacking stretching power (high "P" and small "L"). Curve "C" has been furnished by a flour which gives a soft and runny dough (small "P" and great "L") and is as unsuitable for bread-making as is the flour represented by curve "B." It is readily apparent, however, that if two wheats which gave flours that yielded the unsatisfactory curves "B" and "C" were blended in equal proportions, the resulting mixture would produce a flour similar to curve "A," which would possess satisfactory protein characteristics.

When the Alveograph is used to assess the protein quality of wheat, it is customary to report only two figures, those for stability and strength. Tests on thousands of wheats have shown, however, that if a stability figure of a wheat is 15 to 25 units greater than the strength figure, then the protein of the wheat is satisfactorily balanced. If the numerical superiority of the stability figure over the strength figure is appreciably greater than 25 units, then the protein is inclined to stiffness with a lack of stretching power, while, if the stability figure exceeds the strength figure by much less than 15 units, then the protein is likely to exhibit rather marked extensibility. These points are illustrated in Table III which gives the normal ranges of

TABLE III

RANGES OF STABILITY AND STRENGTH FIGURES OF VARIOUS WHEATS

Wheat	Stability	Strength
Manitoban . . .	80–110	65–105
Garnet	100–125	60–75
Canadian Durum . .	65–90	25–40
Hard Winter . . .	60–90	40–70
Russian . . .	50–85	30–65
Rosafe Plate . . .	60–80	30–50
Barusso Plate . .	65–95	55–70
Australian . . .	25–55	15–35
Indian	70–95	25–35
Danube Basin Wheats .	30–65	25–40
Soft White Pacific . .	25–45	15–25
English . . .	25–55	10–25

stability and strength figures for different types of wheats. Canadian Durum and the Danube Basin wheats, for example, are similar in strength (range 25 to 40) but whereas the stability figures of the latter usually lie between 30 and 65, those of the Durums range from 65 to 90, and the latter wheats are notorious for the stiffness and deadness which characterises the doughs made from flour milled from them. Garnet wheat provides another example; flour from this wheat gives a particularly stiff dough deficient in stretching power and this is reflected in the 40 units by which the stability figure often exceeds the strength figure.

A. J. A.

FLOUR TESTING

TESTS applied to commercially milled flours should serve two purposes—they should reveal the efficiency with which the milling operations have been performed, and the suitability of a flour for the purpose for which it is intended. Tests which fall within the former category are determinations of moisture and ash contents, of brightness and of degree of bleach, while the latter aspect of flour quality is covered by determinations of protein content and diastatic activity and a critical baking test or an instrumental dough test.

1. Moisture Content

If, as is usual, a drying method is employed, the figure obtained will vary according to the temperature and duration of the heating operation.

A widely used method is to heat a 5 gm. sample for 5 hours at 100° C. but quicker methods (heating for 1 hour at 130° C. or for 15 minutes at 155° C.) are available. When comparisons are to be made, all the figures must be obtained by the same procedure or adjusted by appropriate factors. In general, the moisture content of flour is in the region 13·0 to 15·0 per cent.

2. Ash Content

The ash content of flour which contains no mineral additions is an index of the extent to which the flour is contaminated with bran powder, and hence of the grade of the flour, because the ash content of the skins of the wheat grain is in the region of thirty times as great as the ash of pure endosperm. When mineral matter has been added to the flour, and all flour now contains a statutory addition of 14 oz. of *creta praeparata* per 280 lb., the ash content loses its diagnostic value, unless the proportion of added mineral matter *in the sample used in the ash determination* is known.

A suitable method of determining the ash content of flour is as follows:

Weigh 5 gm. of the flour into a tared silica or platinum dish and place in a muffle furnace maintained at a temperature of 600° C. When a white ash has been obtained (usually after 2½ to 3 hours), remove the dish to a desiccator and, when it is cool, weigh it quickly.

Typical ash contents of different types of flour in the absence of mineral additions are given in Table I.

TABLE I

ASH CONTENTS OF VARIOUS TYPES OF FLOUR CONTAINING NO MINERAL ADDITIONS

	Ash Content
Patent flour . .	0·3%–0·4%
Straight run flour of 72 per cent extraction	0·4%–0·5%
Straight run flour of 80 per cent extraction	0·55%–0·65%
Straight run flour of 85 per cent extraction	0·7%–0·9%
Wholemeal . . .	1·4%–1·6%

3. Grade Colour

There are two aspects to the colour of flour—brightness and whiteness. The brightness is determined by the relative proportions of endosperm and bran powder in the flour; the higher the proportion of bran powder the duller the flour. An evaluation of the brightness of a flour, therefore, provides a measure of the extent to which the inclusion of bran powder has been avoided during the milling process, i.e., of the efficiency of the milling operations. The brightness of flour can be reliably evaluated on a numerical basis by an instrument known as the Flour Colour Grader (Fig. 1). This instrument measures by means of photoelectric cells the reflectance of the surface of a suspension of the flour in terms of the reflectance of a standard white surface. The use of a suspension prevents differences in granularity from affecting the readings, while the use of filters prevents serious interference from the degree of bleach on the flour, i.e., from the degree of yellowness. Flour Colour Grader readings of different types of flour are given in Table II.

Fig. 1.—
Flour Colour Grader.
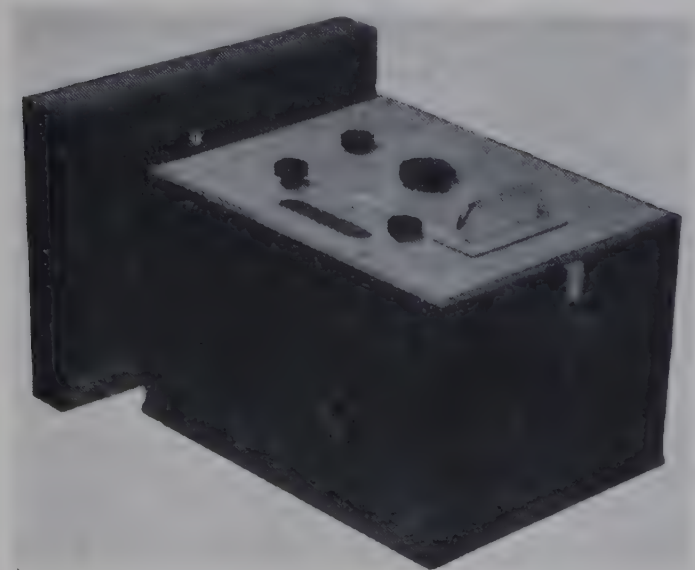
TABLE II

Flour Colour Grader Readings, i.e., Brightness Measurements of Different Types of Flour

	Grade Colour
Patent flour . .	1·5 or less
Straight run flours of 72 per cent extraction	2·0–3·5
Straight run flours of 80 per cent extraction	4·5–6·0

4. Degree of Bleach

The amount of flour milled in Britain which is not artificially bleached by the miller represents a very small proportion of the total output, and most of it is produced for purposes other than breadmaking. A determination of the degree of bleach is consequently a desirable feature in a scheme of flour testing because it provides a check upon the regularity with which the bleaching operation is being performed. Some variation in the whiteness of the flour produced by any one mill is to be expected because the degree of bleach on a flour depends not only upon the dosage of bleaching agent added but also upon the amount of yellow colouring matter natural to the unbleached flour, and this varies to some extent according to the wheats employed. Fluctuations in whiteness due to this factor and to the supplementary effect of atmospheric bleaching subsequent to manufacture are, however, small, and a marked departure from

the usual standard of whiteness would suggest a fault in the application of chemical bleach.

The degree of bleach on a flour is assessed by obtaining a measure of the amount of residual unoxidised yellow colouring matters in the flour and this is accomplished by extracting these unchanged colouring matters in a suitable solvent and measuring the intensity of the yellow colour of the resulting solution. Details of a suitable procedure are:

Weigh 12 gm. of flour into a dry stoppered 4 oz. bottle, add 67 ml. of a 1:1 mixture of petrol and benzol and shake well. Allow to stand overnight and then filter through a No. 5 Whatman paper, discarding the first few ml. of filtrate. Keep the funnel covered during the filtration to reduce evaporation. Measure the absorbency of the filtrate against the pure solvent in a Spekker absorptiometer, using firstly blue filters (No. 7) and then red filters (No. 1). Subtract the absorbency reading obtained with the red filter from the blue filter reading and convert the difference to the corresponding empirical "bleach figure" by means of Table III.

TABLE III

CONVERSION OF SPEKKER ABSORPTIOMETER READINGS OF FLOUR EXTRACTS TO "BLEACH FIGURES"

Absorptiometer Reading	Bleach Figure
0·000–0·030	0·5
0·031–0·050	1·0
0·051–0·070	1·5
0·071–0·085	2·0
0·086–0·100	2·5
0·101–0·115	3·0
0·116–0·130	3·5
0·131–0·140	4·0
0·141–0·150	4·5
0·151–0·160	5·0
0·161–0·170	5·5
0·171–0·180	6·0
0·181–0·190	6·5
0·191–0·200	7·0
0·201–0·210	7·5
0·211–0·220	8·0
0·221–0·230	8·5
0·231–0·235	9·0

Unbleached flours normally give bleach figures of 8·0 or more according to the wheats from which they have been milled and the age of the flours. Lightly bleached flours give figures in the region of 6·0, moderately bleached flours in the region of 4·0, and strongly bleached flours in the region of 2·0.

5. Protein Content

This is calculated from the nitrogen content of the flour by multiplying by 5·7. The nitrogen content is determined by the procedure used for wheat (*see* p. 212) using a 1 gm. sample.

The protein content of flour is less than that of the wheat from which it is milled by 0·5 to 1 units per cent. Bread flours should contain 10 to 12 per cent protein depending on the grade, but figures of 8·5 to 10 per cent are suitable for confectionery flours. Biscuit flours may contain 8·5 per cent protein but lower figures are an advantage.

6. Diastatic Activity—Maltose Figure

The determination of the "maltose figure," which is a measure of the diastatic activity, of flour has been described in the section on "Wheat Testing" (*see* p. 213). The maltose figure of a bread flour should be in the region of 1·7 to 2·0, although satisfactory bread can be obtained when the figure is below or above this range. A figure of 1·5 or less, however, offers little margin of safety against insufficient gas production during fermentation, and when the maltose figure is at this level, the addition of a diastatic malt flour, in a proportion of, say, $\frac{1}{2}$ lb. to 280 lb. of flour, is indicated. A figure significantly above 2·3 is a warning that the alpha-amylase activity of the flour may be unduly great and may give rise to stickiness in the crumb of the bread. There is no cure for this other than a suitable alteration of the wheat blend.

There is no lower limit for the maltose figures of flours intended for the production of goods which are aerated by means other than yeast fermentation, such as, cake and self-raising flours. A high maltose figure, i.e., one significantly above 2·3, may, however, be undesirable and certainly is in self-raising flour, because it can give rise to doughy streaks if the flour is used to produce boiled goods. A low figure is usually preferred in

biscuit flour because otherwise some of the goods may take on too much colour during cooking.

7. Baking Test

While mechanical dough tests are to be recommended for the assessment of the protein qualities of wheat, they should be replaced by a critical baking test when commercial bread flours are under examination.   The reason is that where baking flours are concerned the effects of yeast fermentation, enzymic action and dough manipulation must be taken into account, and this can be done most reliably by submitting the flour to the treatment it will receiver from the baker.   Where baking flours are more restricted in type, as in North America, there is scope for a closely standardised, small scale, laboratory baking test in which the evaluation is based upon loaf volume, but this procedure is not suited to conditions in Britain, where many types of wheat are used in the bread grist and flours vary much more widely in nature.

The type of baking test to be recommended is one in which a 2 lb. loaf is made under controlled conditions and close attention paid by the operator to the properties and behaviour of the dough throughout fermentation.   It is best if the test be performed by a baker.   If this is not possible then the laboratory worker responsible for the test should receive tuition from a baker and obtain first hand knowledge of commercial practice.   Factors to which the test baker should pay attention are the amount of water required to give a dough of the proper consistency; the stability and elasticity of the dough; the gassing power of the dough; oven development; crust and crumb colours; and the general external and internal characteristics of the loaf.   A suitable test baking procedure is as follows:

Weigh 655 gm. of flour into a bowl, place 8 gm. of salt on top of the flour at one side of the bowl and make a bay in the middle of the flour. Place in the bottom of the bay 12 gm. of compressed yeast.   Bring a quantity of water to a temperature in degrees Fahrenheit which is numerically equal to the difference between the temperature of the flour in degree Fahrenheit and 160, and then pour 350 ml. of this water into the bay in the flour.   Disperse the yeast in the water by manipulation with the fingers and then gradually mix in the flour until a dough is formed.   If the dough feels too tight, add 5 to 10 ml. of water during the mixing.   Cover the bowl and place it in a cabinet maintained at 80° F.

After 1½ hours give the dough a thorough kneading, re-cover and place back in the cabinet for a further ½ hour. Then re-knead the dough, shape into a ball and allow it to stand under a cloth on the moulding table for 10 minutes. Mould the dough into the desired shape (a cylinder, if a tin loaf is to be made) and place in a greased baking tin. Place the tin in a cabinet maintained at about 85° F. in which the air has a relative humidity of about 90 per cent, and allow it to remain there for 35 to 45 minutes. Then transfer the tin to a baking oven at a temperature of 450° F. to 500° F. and bake for 40 to 50 minutes. The crust should be brushed over with water about 5 minutes before the loaf is withdrawn from the oven.

SELF-RAISING FLOUR

Self-raising flour is flour to which has been added chemicals which, in the presence of water, and particularly when heated, liberate carbon dioxide. The aeration of a dough made from such flour is brought about by the released carbon dioxide and hence no prolonged fermentation is required as in the case of bread production. A weaker and less proteinous flour than a bread flour is required for self-raising purposes; a protein content of 9 to 10 per cent is ample and a strength figure of 25 to 35, as compared with one of 45 to 60 for bread flours, is sufficient. The moisture content should not exceed 14 per cent if risk of undue loss of CO_2 during storage is to be avoided. The maltose figure is of little significance because the aeration does not depend upon the fermentation of sugar but it can cause trouble if it is high, say, above 2·3. At this level it may cause the formation of dough-like streaks in boiled goods.

The aerating chemicals used in the production of self-raising flour are sodium bicarbonate and an acid phosphate. This latter may be acid calcium phosphate or acid sodium pyrophosphate. The bicarbonate is normally present in a proportion of 3 lb. 4 oz. per 280 lb. of flour, and this calls for the use of 4⅓ lb. of either acid calcium phosphate or acid sodium pyrophosphate. The latter acid ingredient is available, however, in a diluted form which is used with bicarbonate in a ratio of 2:1.

The analysis of self-raising flours follows the scheme employed for bread flours except that often the ash determination is omitted. The only amendment of technique called for is the use of a buffer solution (22 gm. of disodium hydrogen phosphate and 15·4 gm. of citric acid in 1 litre) in the place of water in the preparation of the

suspension for the use in the determination of grade colour. Additional tests are necessary, however, because self-raising flours are required by law to contain not less than 0·40 per cent of available carbon dioxide. The available carbon dioxide content is arrived at indirectly from determinations of the total and residual carbon dioxide contents and the procedures for these determinations are given in the relevant Order. The prescribed methods are:

"The available carbon dioxide shall be determined by ascertaining the difference between the total carbon dioxide and the residual carbon dioxide; and the total carbon dioxide and the residual carbon dioxide shall respectively be determined in the following manner:

(*a*) TOTAL CARBON DIOXIDE : Shall be determined by ascertaining the weight thereof evolved when the self-raising flour is treated with excess of dilute sulphuric acid, the evolution being completed either by boiling for five minutes or by means of reduced pressure.

(*b*) RESIDUAL CARBON DIOXIDE: Shall be determined by taking not less than five grams of the self-raising flour, which shall be mixed to a smooth paste with distilled water, and a further quantity of distilled water amounting in all to not less than twenty times the weight of the flour shall then be incorporated. The liquid shall be heated in a boiling water bath for thirty minutes, being vigorously stirred for the first five minutes and thereafter for approximately half a minute at intervals of approximately five minutes. The liquid shall forthwith be boiled for three minutes, being vigorously stirred during the whole of such period, and then transferred to an apparatus for determining carbon dioxide, through which carbon dioxide-free air shall be passed for not less than ten minutes. The residual carbon dioxide is the weight thereof evolved when the self-raising flour so treated is further treated with excess of dilute sulphuric acid, the evolution being completed either by boiling for five minutes or by means of reduced pressure."

The chemical analysis of self-raising flour is supplemented by a baking test. It is usual to make a round of scones from the flour and to appraise the volume, crumb texture and crumb colour of the goods. A suitable baking formula is:

Rub 1 oz. of fat into 4 ozs. of the flour and then make a bay in the mixture. Weigh 1 oz. of sugar into the bay. Pour about 90 ml. of milk into the bay and stir the sugar with the fingers until it is dissolved. Then work the flour in until a slack dough is obtained. Mould this into a ball, flatten with the hand and cut into four quadrants. Place on a greased tin and bake at about 420° F. for 10 to 15 minutes.

Another test which is applied to self-raising flour, more particularly when the maltose figure is high, is to make from it a boiled dumpling. If the self-raising flour is satisfactory, the cut dumpling should have a dry, mealy texture which shows no doughy streaks. If the cut surface is sticky, or dough-like streaks are present, the flour cannot be regarded as satisfactory. Dumplings can be made as follows:

Prepare a fairly slack dough from the flour and make it into a ball. Tie this into a cloth, drop it into boiling water and allow it to remain there for 20 minutes.

BISCUIT FLOURS

The chemical tests applied to biscuit flours are the same as those used in the examination of bread flours. The protein content should be low—8 per cent or less—and a low maltose figure is also desirable. The protein characteristics are of particular importance and should be studied on a mechanical dough testing instrument. They cannot be satisfactorily assessed by means of a yeast baking test. If the Alveographe is used, a satisfactory biscuit flour will have a stability not greater than 35 and a strength not above 20; within reason the lower the stability and the greater the extensibility the better the flour for biscuit work.

SPECIAL FLOUR TESTS

Rope Spore Count

Bacteria of the *B. mesentericus*—*B. subtilis* group are found on wheat and during the milling find their way into the flour. These micro-organisms form spores, which survive the baking process and are thus present in bread (see Fig. 2). If bread is kept warm and moist, the spores are transformed into bacteria and these then multiply and in so doing cause the crumb of the bread to assume a yellowish-brown colour and a sickly smell and eventually to become very sticky. This bacteriological disease of bread is called "rope" because the sticky crumb can be pulled out into rope-like threads.

Whether rope develops in bread which is stored under conditions favourable to the development of the causative bacteria is

determined mainly by the conditions under which the bread has been fermented, baked and cooled; a vigorous fermentation, thorough baking and rapid and thorough cooling reduce the risk of the disease. Other factors being equal, however, the greater the number of rope spores present in a flour the greater the
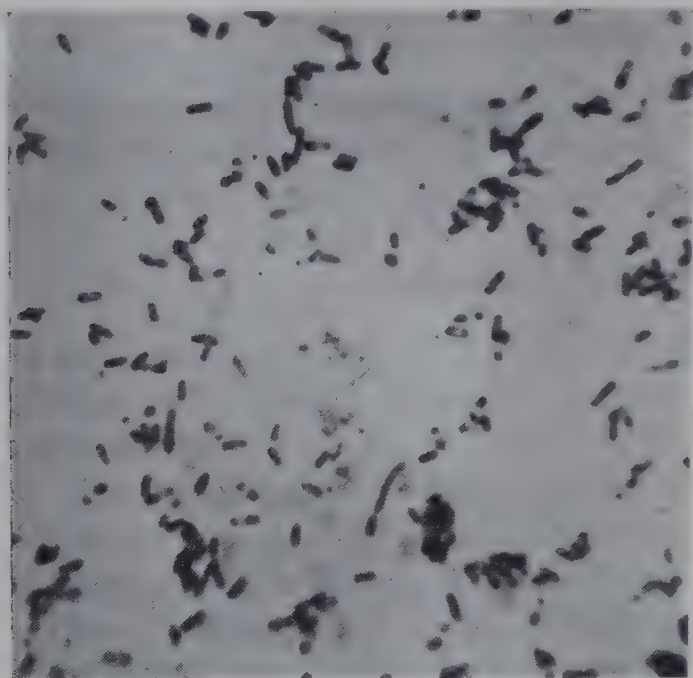
FIG. 2.—BACTERIA AND
ROPE SPORES.

likelihood of bread made from that flour becoming ropy. When a case of rope occurs, therefore, a rope spore count on the flour may be needed. This can be made by the procedure which follows:

Measure 100 ml. of sterile 0·5 per cent sodium chloride solution into a sterile 8 oz. wide-mouthed glass-stoppered bottle and 10 ml. quantities into each of nine sterile 2 oz. glass-stoppered bottles. To the solution in the 8 oz. bottle add 10 gm. freshly ignited sand. Weigh 32 gm. of the flour on to a sterile watch glass and transfer to the 8 oz. bottle and shake vigorously for 2 minutes. Transfer 10 ml. of the resulting solution by means of a sterile pipette with a filed off tip to one of the bottles containing 10 ml. of saline and shake the bottle for at least 1 minute.

Transfer 10 ml. of this dilution by means of a sterile pipette to another of the bottles containing 10 ml. of saline and shake. Repeat this procedure until the nine small bottles contain a series of dilutions, each of which has half the concentration of the preceding one. Then add by means of a sterile pipette 1 ml. of each of the suspensions, i.e., that in the large bottle and those in the small bottles, to tubes of nutrient broth. Mix the broth and the suspension by rotating each tube between the palms of the hands and then heat the tubes in boiling

water for 20 to 30 minutes. Again mix the contents of the tubes and incubate at 37° C. for 48 hours. At the end of 24 hours re-mix the contents of those tubes which show no pellicle formation. At the end of 48 hours count as positive those tubes which exhibit a typical pellicle on the surface of the broth.

The average number, N, of sterile tubes in the series of ten concentration levels employed is obtained by dividing the total number of sterile tubes by five, since five replicates have been used at each concentration level. Reference to Table IV will

TABLE IV

RELATION BETWEEN R, THE NUMBER OF ROPE SPORES IN 1 ML. OF PRIMARY SUSPENSION AND N, THE AVERAGE NUMBER OF STERILE TUBES

N	R	N	R	N	R	N	R
0							
0·1	1,224	2·6	73·9	5·1	12·05	7·6	2·08
0·2	907	2·7	68·5	5·2	11·23	7·7	1·931
0·3	732	2·8	63 5	5·3	10·47	7·8	1·793
0·4	615	2·9	58·9	5·4	9·75	7·9	1·662
0·5	528	3·0	54·6	5·5	9·09	8·0	1·539
0·6	461	3·1	50·7	5·6	8·48	8·1	1·423
0·7	406	3·2	47·1	5·7	7·90	8·2	1·312
0·8	362	3·3	43·8	5·8	7·37	8·3	1·208
0·9	324	3·4	40·7	5·9	6·87	8·4	1·108
1·0	291	3·5	37·8	6·0	6·41	8·5	1·014
1·1	263	3·6	35·1	6·1	5·98	8·6	0·924
1·2	239	3·7	32·7	6·2	5·57	8·7	0·838
1·3	217	3·8	30·4	6·3	5·20	8·8	0·757
1·4	198·2	3·9	28·3	6·4	4·85	8·9	0·679
1·5	181·2	4·0	26·3	6·5	4·52	9·0	0·604
1·6	166·0	4·1	24·5	6·6	4·21	9·1	0·532
1·7	152·3	4·2	22·8	6·7	3·93	9·2	0·464
1·8	139·9	4·3	21·2	6·8	3·67	9·3	0·398
1·9	128·8	4·4	19·77	6·9	3·42	9·4	0·334
2·0	118·6	4·5	18·42	7·0	3·19	9·5	0·273
2·1	109·4	4·6	17·16	7·1	2·97	9·6	0·215
2·2	101·0	4·7	15·98	7·2	2·77	9·7	0·1581
2·3	93·3	4·8	14·89	7·3	2·58	9·8	0·1036
2·4	86·3	4·9	13·87	7·4	2·40	9·9	0·0509
2·5	79·9	5·0	12·93	7·5	2·23	10·0	0

reveal what number of organisms, R, are required in 1 ml. of the most concentrated suspension of the series in order to give rise to N sterile tubes. This number R divided by 0·32 will give the number of rope spores per gram of the flour.

The rope spores in normal flours amount to only a few per gram but higher figures may be encountered in low grade flours or in flours made from unwashed wheats.

Vitamin B₁

Because of the adoption of nutritive standards for flour, the cereal chemist may be called upon to check the nutrient content of either natural or "restored" flour. The two vitamins for which standards are in force are vitamin B_1 and nicotinic acid and methods for assaying these nutrients are available. The determination of the amount of vitamin B_1 in flour is a relatively simple procedure but assays of nicotinic acid are more difficult. The best and most reliable method of assaying the nicotinic acid content of flour is a microbiological one but this takes several days to complete and calls for a laboratory equipped for and an operator experienced in bacteriological procedures. In most instances, however, a determination of B_1 suffices, because if this is present in correct amount the nicotinic acid content is not likely to be seriously at fault.

The chemical determination of vitamin B_1 in flour involves the extraction of the vitamin with acid, the oxidation of the vitamin to thiochrome, and the measurement of the concentration of the thiochrome by means of the blue fluorescence it gives in ultra-violet light. The details of the procedure are as follows:

Weigh out into conical flasks two 20 gm. portions of the flour. To one of them add 100 ml. of dilute hydrochloric acid (2·5 per cent by volume) and to the other add 90 ml. of the same acid and 10 ml. of a solution of vitamin B_1 in hydrochloric acid of the same strength having a concentration of 2 i.u. per ml. Shake the flasks and allow them to stand, with an occasional shaking, for two hours. Then centrifuge the contents of the two flasks.

From each of the two clear supernatant liquors remove by pipette two aliquots of 3 ml. and run each of them into a test tube. To each tube add 2 ml. of methyl alcohol and then lead a stream of air into the mixture so as to keep it in a state of agitation. To one of each pair of tubes containing the same supernatant liquor add a mixture of 13 ml. of isobutanol, 2 ml. of a 30 per cent solution of caustic soda and 2 ml. of an 0·9 per cent solution of potassium ferricyanide, and to the other

tube of each pair add a mixture of 13 ml. of isobutanol, 2 ml. of a 30 per cent solution of caustic soda and 2 ml. of distilled water. Three minutes after the addition of these mixtures stop the stream of air and centrifuge the contents of the four tubes.

Remove from each centrifuge tube 10 ml. of the supernatant liquor and run into a dry test tube containing 1 ml. of ethyl alcohol. Pour the contents of each tube in turn into a test tube chosen for its low fluorescence and note the galvanometer deflection obtained when the tube and its contents are placed in a fluorimeter.

Let T_b be the reading given by the unfortified flour extract not treated with potassium ferricyanide

T_o be the reading given by the unfortified flour extract treated with potassium ferricyanide

S_b be the reading given by the flour extract fortified with added vitamin B_1 but not treated with potassium ferricyanide

S_o be the reading given by the flour extract fortified with added vitamin B_1 and treated with potassium ferricyanide

Then the vitamin B_1 content of the flour in i.u./g. is given by:

$$\frac{(T_o - T_b)}{(S_o - S_b) - (T_o - T_b)}$$

Iron

The nutritive status prescribed for flour includes a standard for iron, and hence determinations of the proportion of this nutrient in flour may be required. A suitable method is as follows:

Ash 10 gm. of flour until free from carbon and then dissolve by heating with 4 ml. of 50 per cent hydrochloric acid on a water bath. If the flour being tested has been restored with *ferrum redactum,* the heating may have to be continued for several hours before solution is complete. Filter the solution through an acid-washed hardened filter paper and rinse the dish several times with hot water containing a few drops of hydrochloric acid. Wash the filter paper with hot water. To an aliquot of the filtrate add 10 ml. of a 20 per cent solution of citric acid and two drops of thioglycollic acid. Add ammonia (sp.gr. 0·880) to the liquid until it turns red and then add a further 0·5 ml. Make the liquid up to 100 ml., first removing any precipitate that has formed, and determine the absorbency on the Spekker absorptiometer using the green filter (No. 5). Subtract from this a "blank" reading which is obtained by performing the operations just described with the omission of the flour. The reading of the test solution corrected for the blank is converted into iron content by means of a calibration curve. This is constructed by plotting Spekker readings against known concentrations of iron after they have been corrected by a "blank" reading provided by the reagents without the iron.

Q

Creta Praeparata

Several methods are available for determining the amount of *creta praeparata* in flour, some depending upon the determination of calcium and others on the determination of carbon dioxide. The method to be described is in the former category. It is satisfactory for checking the *creta* content of ordinary flour but it could not be used if the flour contained any other form of calcium. Details of the method are:

Ash 5 or 10 gm. of flour and dissolve the ash in 3 ml. of concentrated hydrochloric acid. Dilute the solution to 100 ml., add 3 gm. of citric acid, make alkaline with ammonia and then bring back to a slight acidity with hydrochloric acid. Boil the solution and add 30 ml. of a 3 per cent solution of ammonium oxalate drop by drop. Allow the precipitate to settle overnight and then filter on a sintered glass crucible (porosity G3 or G4). Wash the precipitate with a 1 per cent solution of ammonium oxalate and then five times with cold water. Wash the bulk of the precipitate into a beaker and dissolve the remainder of the precipitate by filling the paper with hot 20 per cent sulphuric acid and then washing with hot water. Add the sulphuric acid and the washings to the beaker containing the precipitate, warm to 70° C. to 80° C., and titrate with N/10 potassium permanganate. Each ml. of N/10 potassium permanganate corresponds to 0·002 gm. of calcium.

ANALYSIS OF MILL BY-PRODUCTS

The main by-products of flour milling—bran and sharps or Weatings—are used as animal feeding stuffs. The analysis to which these materials are submitted relates, therefore, to their feeding value, and more particularly to those constituents for which the law requires a declaration to be made at the time of sale. The tests commonly applied are determinations of protein, oil and fibre.

Protein Content.

The protein content is arrived at by determining the nitrogen content and multiplying by the factor laid down in the Fertilisers and Feeding Stuffs Act 1932, namely, 6·25. The nitrogen content can be determined by the method described under "Wheat Testing" (*see* p. 212) but the method prescribed in the Act is as follows:

" A weighed portion of the sample shall be transferred to a Kjeldahl digestion flask, 25 millilitres of concentrated sulphuric acid (or more if necessary) shall be added and the flask gently heated until frothing

ceases. Ten grams of potassium or sodium sulphate (anhydrous) shall then be added and the flask further heated until the colour of the clear liquid ceases to diminish. The heating shall be continued for an hour thereafter to ensure complete oxidation of the organic matter. The operation may be accelerated by the addition of a small crystal of copper sulphate or a globule of mercury to the liquid in the digestion flask.

The quantity of ammonia present in the liquid shall be determined by distillation into standard acid after liberation with alkali and, where mercury has been used, with the addition also of sodium or potassium sulphide solution.

The materials used shall be examined as to their freedom from nitrogen by means of a control experiment carried out under similar conditions with the same quantities of the reagents which have been employed in the actual analysis, one gram of pure sugar being used in place of the weighed portion of the sample. The quantity of standard acid found to have been neutralised in this control experiment shall be deducted from the total quantity of acid neutralised in the distillation of the sample."

The methods prescribed in the Fertilisers and Feeding Stuffs Act 1932 for the determination of oil and fibre are as follows:

Oil Content

"A weighed quantity of the sample shall be placed in an extraction thimble, which shall then be placed in an extraction apparatus and extracted with petroleum spirit b. pt. 40–60° C. At the end of three to four hours the thimble shall be removed from the apparatus, dried and its contents finely ground, preferably with sand, in a small mortar previously rinsed with petroleum spirit. The substance shall then be returned to the thimble, the mortar being washed out with petroleum spirit, and the extraction continued for another hour. The extract should be free from suspended matter. After evaporation of the solvent, the oil shall be dried at 100° C. and weighed.

In the case of samples containing saccharine matter, the weighed portion in the thimble shall be washed with water and then dried, previous to the extraction.

Fibre Content

"Two or three grams, accurately weighed, shall be extracted with petroleum spirit b. pt. 40–60° C. in an extraction apparatus, or at least three times by stirring, settling and decantation, and the dry residue transferred to a conical 1000 millilitre flask. The material must not be further ground during extraction. A volume of 200 millilitres of a solution containing 1·25 gm. of sulphuric acid (H_2SO_4) per 100 millilitres measured at ordinary temperature and brought to boiling point, shall be added to the flask and heated. The contents of the flask must come to boiling within 1 minute and the boiling throughout

must be gentle and continuous for exactly 30 minutes, the original volume being maintained.

The flask shall be rotated every few minutes in order to mix the contents and remove particles from the sides. At the end of 30 minutes the flask shall be removed and the contents poured at once into the shallow layer of hot water remaining in a funnel fitted with a pump-plate or alternatively into the similar layer remaining in a Buchner funnel. The funnel shall be prepared by cutting a piece of cotton cloth or filter paper to cover the holes, so as to serve as a support for a disc of ordinary filter paper; boiling water shall be poured into the funnel and allowed to remain until the funnel is hot, whereupon suction is applied. The experiment shall be discarded if the time of filtration of the bulk of the 200 millilitres exceeds 10 minutes. The residue shall be washed with boiling water until the washings are free from acid. The residue shall then be washed from the filter paper back into the flask with a volume of 200 millilitres of a solution of sodium hydroxide, containing 1·25 grams of sodium hydroxide (NaOH) per 100 millilitre free or nearly free from sodium carbonate, measured at ordinary temperature, and brought to boiling point.

The contents of the flask shall be boiled for exactly 30 minutes, the precautions given for the treatment with acid being observed. At the end of 30 minutes the flask shall be removed and its contents immediately filtered through an ordinary filter paper. The residue collected on the filter paper shall be washed with boiling water, then with a solution of 1 per cent hydrochloric acid and again with boiling water until free from acid. The residue shall then be washed twice with 95 per cent alcohol, and three times with ether. The residue shall then be transferred to a dried weighed ashless filter paper, dried at about 100° C. in an oven and weighed in its weighing bottle until constant in weight. The ash of the paper and contents shall be determined by incineration at a dull red heat. The weight of ash shall be subtracted from the increase of weight found on the paper and the difference shall be reported as fibre."

<div align="right">A. J. A.</div>

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BAKERY MATERIALS

FLOUR is by far the most important ingredient used in bread-making and it is the most variable. It is, therefore, desirable that flours should be tested regularly so that the main characters of a flour which affect bread quality should be known before the flour is used.

The most useful data that can be supplied to a baker about flour are:

1. Flour strength
2. Water absorption
3. Colour
4. Flavour
5. Enzymic activity

1. FLOUR STRENGTH

This term is difficult to define and even more difficult to evaluate numerically. It is made up of a number of factors mostly concerned with the physical properties of the proteins of wheat flour after hydration, although the quantity of protein present also has some influence on flour strength. Flour strength is the most important of the factors which influence the type of bread which can be made from wheat flour.

A flour is termed "strong" if, after adequate fermentation, the dough will rise in the oven and give bold loaves of good volume. It is termed "weak" if the bread lacks volume because the dough will not retain the gas which is produced by fermentation. A strong flour will carry a weak flour and so by judicious blending it is possible to make use of both strong and weak flours. The dough from a strong flour is capable of standing up to high speed mixing or to a prolonged fermentation, whereas the dough from a weak flour breaks down under either of these disruptive processes.

Dough Testing Instruments

The above facts are used in one of the best known dough testing machines, the Brabender Farinograph, as a means of measuring flour strength.

The Brabender Farinograph (Fig. 1)

The dough is mixed in a double-bladed mixer and maintained at a constant temperature by water flowing through the jacket. After a preliminary test to find the amount of water required to give a dough of a standard consistency a dough is mixed for 10 to 15 minutes. The time taken for the dough to attain standard consistency, the time it can maintain this consistency and the extent it falls away from it with further mixing give a measure of the strength of the flour. Typical curves are shown for strong and weak flours in Figs. 2 and 3. The Farinograph can also be used for measuring the effect on a dough of fermentation and of added ingredients—in each case the ability of the dough to stand up to the high speed action of the mixer being regarded as a measure of strength.
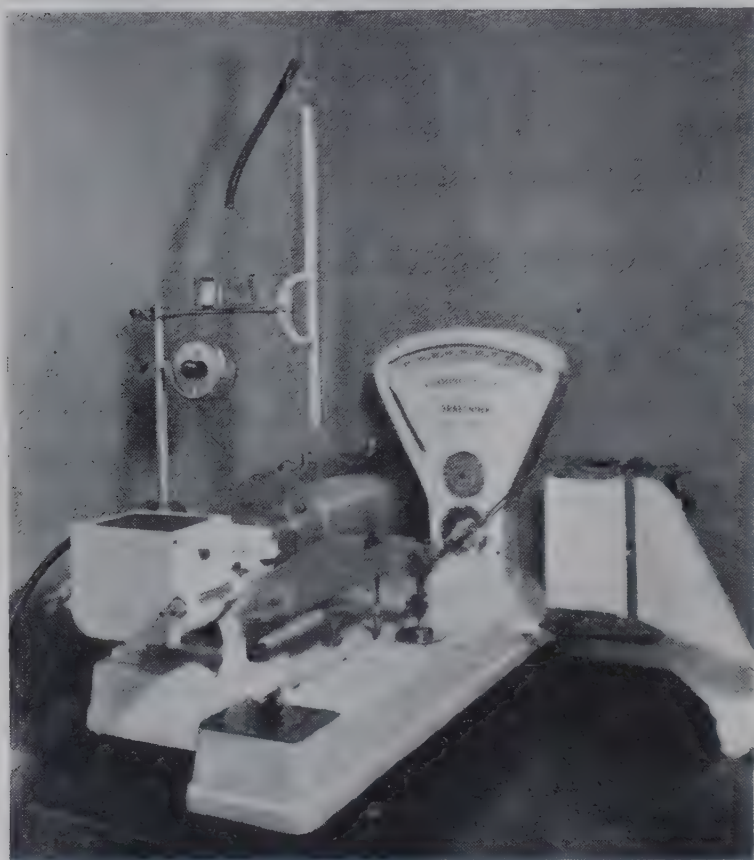
Fig. 1.—Brabender Farinograph.
(*Henry Simon, Ltd.*)

For some purposes, however, the action of the high speed mixer of the Farinograph is too severe and measurements of the resistance and the extensibility of the dough when subjected to a much more gentle stretching action can be more valuable. For this purpose three instruments have been designed and are in common use. These are the Chopin Alveograph, the Extensometer, designed by the Research Association of the British Flour Millers, and the Brabender Extensograph.

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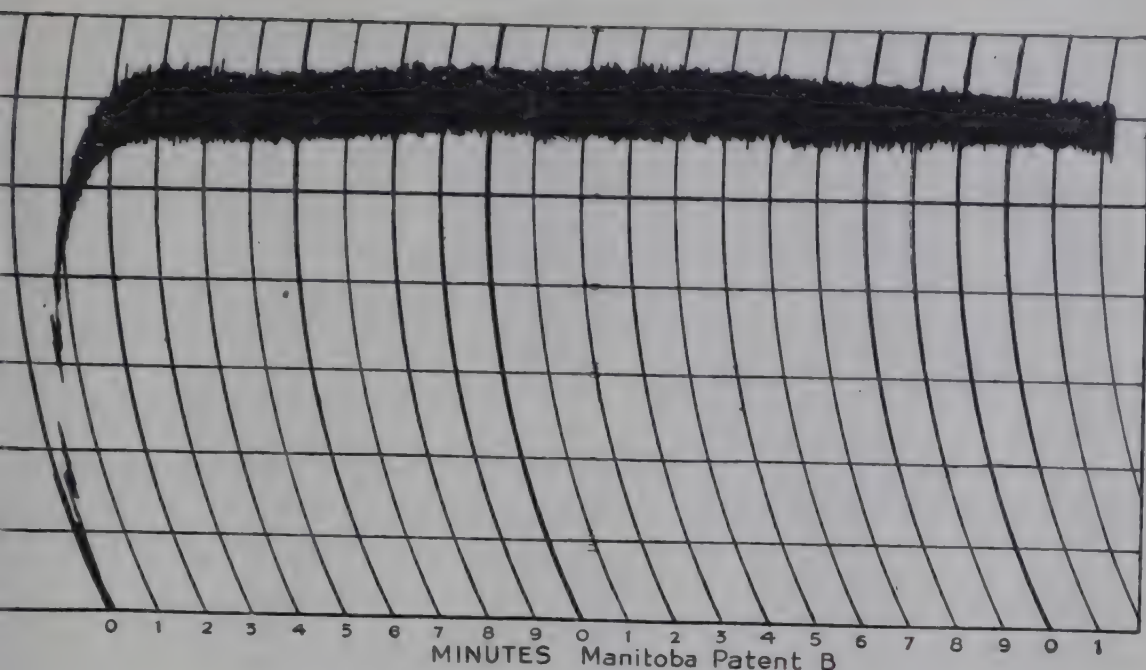
FIGS. 2 and 3.—CURVES
OBTAINED ON THE
BRABENDER FARINOGRAPH
FOR STRONG AND WEAK
FLOURS.
(*The British Arkady Co., Ltd.*)

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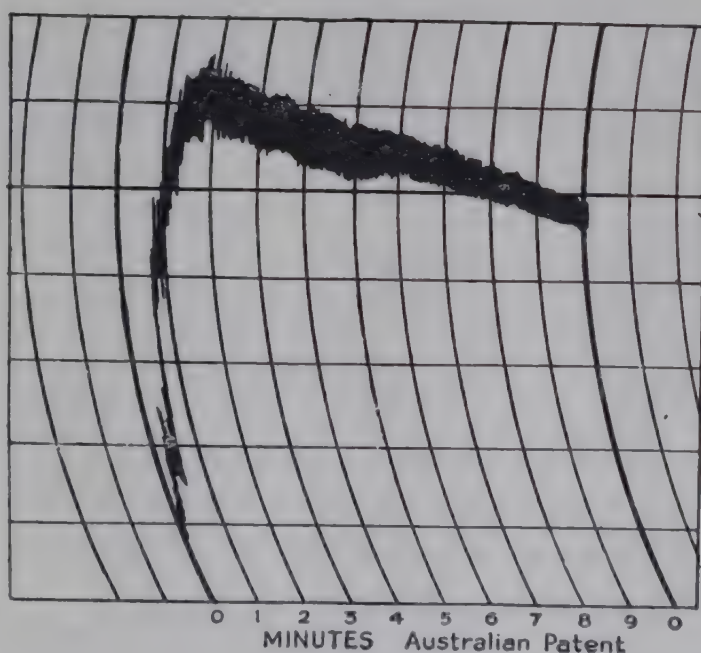
THE CHOPIN ALVEOGRAPH (*see* p. 217).—This instrument
measures the resistance and extensibility of a piece of dough
when blown up under standard conditions to form a bubble
and the other two instruments measure the same factors when a
piece of dough is stretched to breaking point between two arms.
It is not possible in the limits of our space to go into the details
of these tests, or to compare the advantages and disadvantages
of the three instruments.
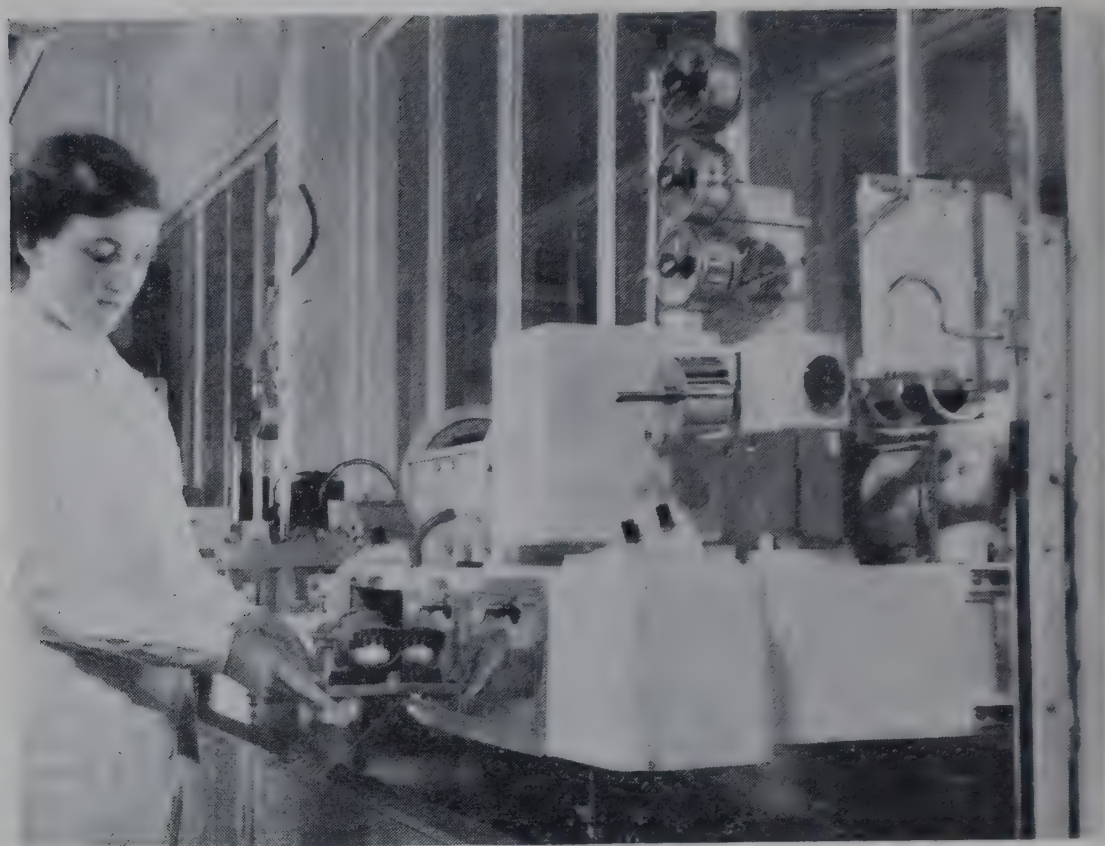
Fig. 4.—Brabender Extensograph.
(*Henry Simon, Ltd.*)

Isolation of Gluten

Quite a useful method of assessing flour strength without the aid of expensive instruments is the older method of washing out gluten from a dough. To be of any use it must be carried out under standardised conditions and even then much depends upon the experience and judgment of the operator. The usual method of carrying out this test is as follows:

A small quantity of flour (25 or 50 gm.) is weighed out into a small kneading machine and made into a stiffish dough with the requisite amount of water. A revolution counter should be fitted to the kneader, so that the same amount of mixing can be given in all cases. The dough, if fairly stiff, will come away cleanly and wholly from the machine; it is then rolled up into a ball, placed into a beaker of water at 70° F. and allowed to stand for an hour. This period allows the gluten to form more definitely from its constituent proteins. The ball is then submerged in successive lots of water at 70° F., being squeezed so as to expel the starch. The wash-water becomes clearer and clearer and the last lot should be free from starch granules. Some operators work in running water, but the successive method is considered the better. After washing, the gluten is worked into a coherent mass and left in water for half an hour before examining.

This examination of gluten is a process incapable of expression on paper. It is purely a question of acquired experience. The operator describes the gluten by such terms as "short," "elastic," "tough," "sticky" and so on, and by consideration of its one or more characteristics is able to form an opinion as to the quality and origin of the flour from which it is derived. After the examination for quality the gluten is spread on a tared filter paper, weighed, dried in an oven to constant weight and weighed again to ascertain its quantity which it is important to know.

2. WATER ABSORPTION

A strong flour will usually absorb 57 to 59 per cent of its own weight of water at doughmaking whereas a weak flour may absorb no more than 50 per cent. Even a 1 per cent difference means nearly three 1-lb. loaves per 280 lb. flour, so extra water absorption affects the yield of bread and hence the value of the flour to the baker. Each of the several dough testing instruments already referred to has a procedure for measuring water absorption of a flour but these figures generally need some correction to bring them into line with practical breadmaking. One reason for this is that when dough is being divided and moulded by machinery the stickiness or tackiness of the dough pieces is as important as the softness or consistency of the dough and it is difficult to devise an instrumental test which will measure both these factors. For this reason an experienced person can often determine a water absorption figure more quickly and more accurately than by other means. The method used is:

56 gm. of flour are weighed into the kneader, 0·8 gm. salt and an average quantity, say, 30 ml. of water added from a burette. This corresponds to 15 gallons to the 280 lb. sack of flour. The mixture is then worked into a dough and the operator feels whether the consistency is too tight or not. If necessary, a little more water is added and worked into the dough until the correct consistency is obtained.

3. FLOUR COLOUR

The colour of flour is important because the British public give preference to bread with a clean bright crumb colour and if the baker uses a flour of poor colour he cannot hope to make bread of the type his customers want. The colour of flour is primarily derived from two sources; the yellow colour of xanthophylls dissolved in the wheat oil and the brownish colour of bran particles. In wheatmeals which may contain 5 to 15 per cent of bran particles the brown colour predominates in the bread,

but even as little as 0·5 to 1 per cent of bran makes a very noticeable difference to the colour of white and National flours.

The yellow colour in wheat flour can be partially bleached by several oxidising agents, including the oxygen in the atmosphere, but these will be referred to later. A measurement of the yellow colour alone can be made after extraction with any oil solvent such as petrol or carbon tetrachloride. The more important colour of the bran particles which largely determines the grade and therefore the price of the flour cannot be bleached out and its measurement is by no means easy. In recent years, however, a Flour Colour Grader has been devised which does measure the darkening effect of the bran particles without being influenced by the yellow colour of the xanthophylls (*see* p. 222).

Flour Bleaching Agents and Improvers

For many years it has been standard practice in flour mills to subject flour to some bleaching process and often the bleaching agent has also had an improving effect due to oxidation of the flour proteins. The first process of bleaching which was generally adopted was the "electric arc" process—the active agent being nitrogen peroxide—but this was superseded by chlorine, nitrogen trichloride (Agene process) and chlorine dioxide (Dyox). These chlorine compounds act as gluten improvers as well as bleachers. Benzoyl peroxide (Novadelox) bleaches the wheat oil slowly but has no oxidising effect on the gluten, and it is useful to millers to have this type of bleaching agent. It is generally mixed with some innocuous filler such as calcium phosphate before being added to flour, as the amount required is quite small—about ½ oz. to 280 lb. of flour.

The underlying idea of bleaching flour is to imitate in home-milled flours the "age-ing" process which takes place by atmospheric oxidation in flours kept in store for several months. Freshly milled flour does not make such good bread as "aged" flour, so without these flour improvers the home miller would be at a disadvantage compared with millers in Australia or Canada.

Not all flour improvers are gases, some are powders, such as acid calcium phosphate, ammonium persulphate and potassium bromate. Calcium phosphate is now seldom used except in Ireland. Qualitative tests for persulphate and bromate treatment are simple.

For persulphate, the flour is made into a soft dough over which is poured a 1 per cent solution of benzidine in alcohol. Blue spots indicate the presence of persulphate. For bromate, a wetted surface of flour is treated with a few drops of an acidified potassium iodide solution. Dark brown spots show up wherever there are particles of bromate.

4. FLAVOUR

Wheat and flour can absorb flavours from other materials and so give rise occasionally to foreign flavours in bread. Scented seeds, timber, apples, or musty wheat have been found to have been the cause of unusual flavours in bread. By scalding a flour paste with boiling water it is often possible to detect such flavours more readily.

Lower grades of flour, e.g., the National flours of 80 and 85 per cent extraction have more of the natural wheat flavour than white flour (70 per cent extraction or less) and in consequence when white bread was once more permitted to be sold in Great Britain in 1952 many people rejected it because it lacked flavour by comparison with the National bread.

5. ENZYMIC ACTIVITY

The enzymes most studied in wheat flour are diastatic enzymes and generally these are sufficiently active to produce from the starch sufficient maltose to provide sugar for the later stages of fermentation and to colour the crust when the bread is baked. If the diastatic enzymes are not sufficiently active they can be reinforced by adding malt flour, or extract, or preparations of fungal enzymes.

If flour contains an excess of diastatic enzymes, too much starch may be attacked during the baking of the bread and the crumb feels doughy and is difficult to slice after the usual period of cooling. This may become a serious problem because no suitable diastase inhibitor has been discovered, so there is no easy way to combat excess of diastase except by blending flours.

Brabender Amylograph

Diastatic activity of flour can be measured by a determination of the maltose produced in a flour suspension under standardised conditions for one hour. This is called the *Maltose Figure*. Another method which may prove more useful is to measure the

viscosity of a flour paste as it is heated up from 25° C. to 90° C. This is done by means of the Brabender Amylograph, a photograph of which is shown in Fig. 5. Because this method follows the same temperature rise which takes place during baking inside a loaf of bread it probably gives a truer picture of diastatic activity than a determination made at a constant temperature at the lower end of the range, such as 27° C. or 30° C.
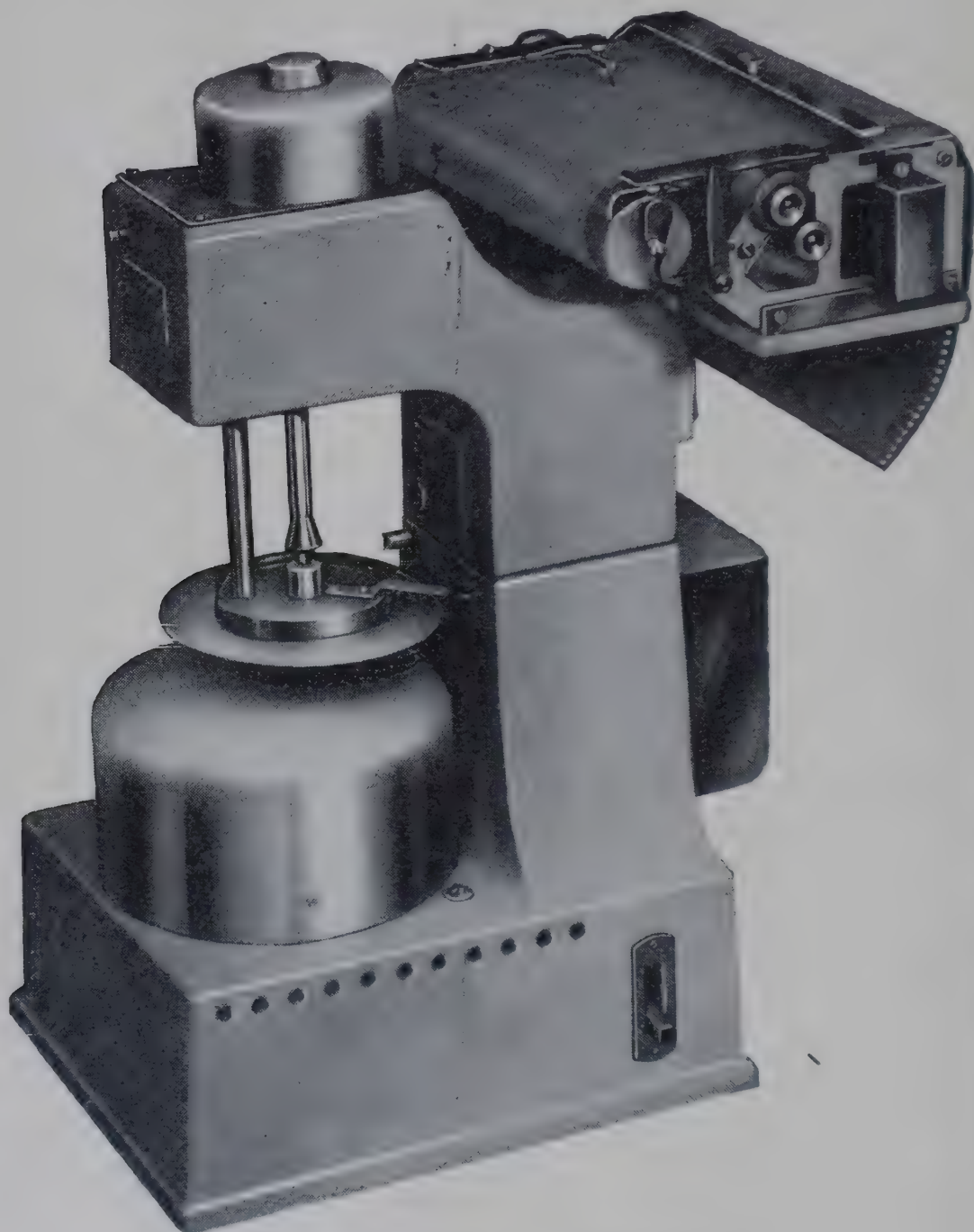
FIG. 5.—THE BRABENDER AMYLOGRAPH.
(Henry Simon, Ltd.)

Proteolytic enzymes in excess are rare but can be very harmful to bread quality. On the other hand, with certain types of flour a small addition of proteases may be quite beneficial. Very little work has yet been done to standardise a test for measuring the activity of proteases which may be useful in breadmaking.

YEAST

The only test carried out as part of the bakery routine is one for activity. Moisture is relatively unimportant; yeast contains normally some 75 per cent of moisture so that 1 or 2 per cent either way is not of tremendous significance, especially as there is no definite standard for it.

Testing for Activity

The testing of yeast for activity is carried out in several ways and none of them is over satisfactory. They are, however, on a level with the gluten-washing test—if a worker of experience always carries out the test, he can draw safe conclusions from the result. Some laboratories test yeast activity by making a dough of standard composition, placing it in standard-sized tins under standard conditions. Others rely on the *Hayduck apparatus*, which has the advantage that many other materials besides yeast can be tested by means of it. Compared with many other scientific instruments, it is crude and elementary, yet it is doubtful whether there is a more useful single piece of apparatus in the cereal laboratory.

The Apparatus Used

The apparatus consists of a thermostatically controlled water-bath in which are placed a series of jars each containing a small piece of dough, each prepared under identical conditions in the little kneader already referred to. The yeast is usually made up into a suspension and added from a pipette.

The jars are each connected with a reservoir containing saturated salt solution (in which carbon dioxide does not dissolve to any extent), or paraffin, and as the dough ferments, the gas evolved drives over the brine into graduated cylinders. The latter are read every half hour or hour according to the conditions used. Great care has to be exercised to ensure there is no leakage.

Some operators use a batter instead of a dough, or even a solution of sugar and yeast nutrients, in which case the gas evolution is greater than with a dough; but there is a great deal to be said for using a dough, because, although the latter is so
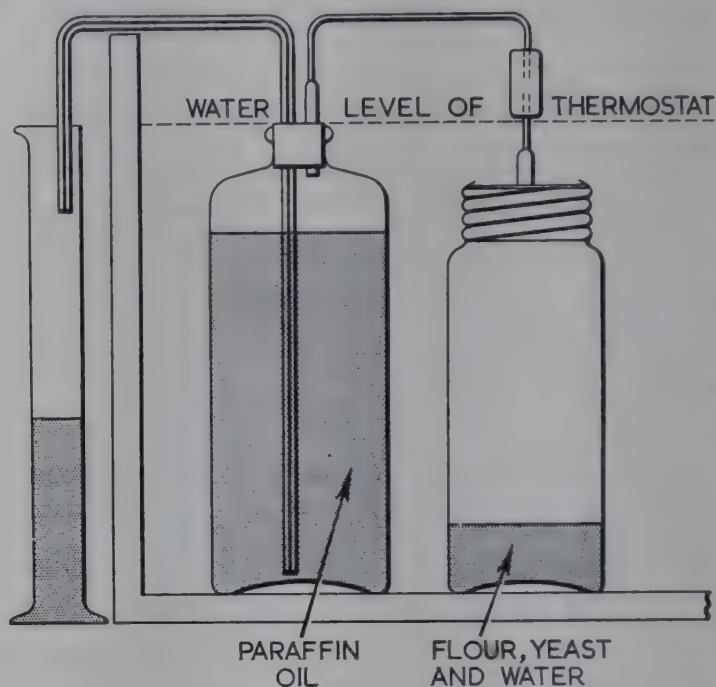
FIG. 6.—APPARATUS USED IN MEASURING VOLUME OF GAS PRODUCED FROM A DOUGH OR BATTER.

The volume of paraffin displaced is measured at frequent intervals.

small as to render it impossible to interpret the results directly into terms of the large masses used in a bakery, yet the yeast is acting under something approaching the conditions it will have in the bakery.

ROPE

Only brief reference can be made to this disease of bread. It is a bacterial infection introduced generally in the flour. There are always rope spores in bread and it is only when climatic conditions are favourable (i.e., high temperatures and humidity) that the danger of spoliation from this cause is serious. Fortunately, rope-spores only develop slowly in an acid medium, and the addition of acid substances is normally a sufficient preventive. (For results of testing see Fig. 7 opposite, and also on p. 229.)

BAKING TEST

Many bakeries have no laboratories and in these cases, recourse must be had to a small baking test. It must be emphasised that bulk action plays an important part in fermentation and that

ROPE BACTERIA PRESENT   ABSENT
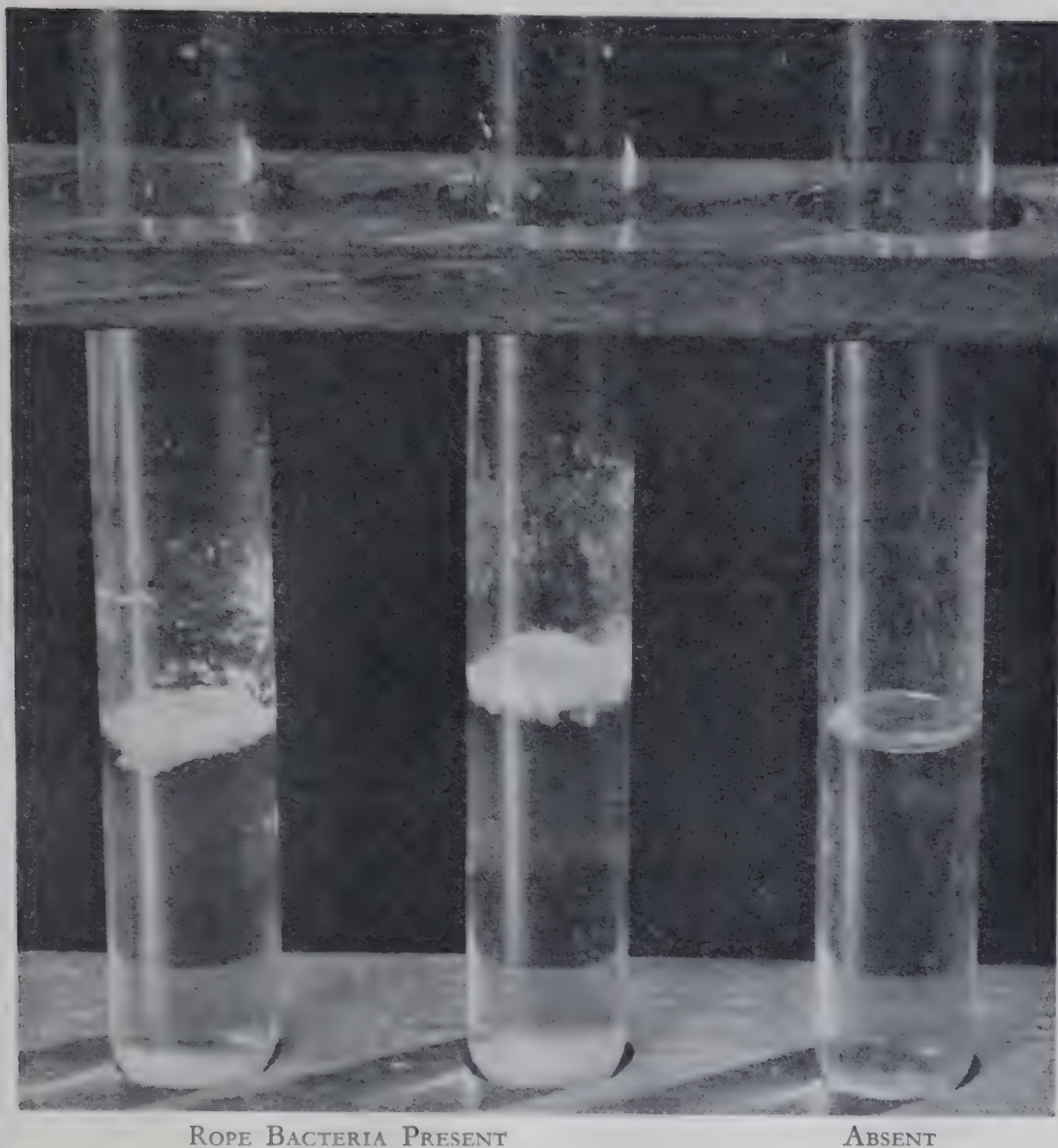
FIG. 7.—SKINS OF ROPE. PRODUCED IN BROTH BY INCUBATION.

(*The British Arkady Co., Ltd.*)

the course of fermentation in a small dough, of say, 7 lb. is quite different from that in a 400 lb. dough, but the experienced baker soon learns what to look for in his small-scale baking test, and is seldom misled. In cereal laboratories, too, a small scale baking test is advisable, if circumstances permit. This should be done for two reasons:

(1) During actual manipulation of the dough and its progress during the fermentation stage, many facts can be learned which are not brought to light by laboratory tests.

(2) A baking test acts as a confirmation of laboratory tests and

it is just as well to have this confirmation.   Although conclusions drawn from the work in the laboratory are definite and sound in the majority of cases, it occasionally happens that a "queer" flour is encountered, which behaves differently in the bakery from what the laboratory report would indicate.

One can read into this last sentence the fact that laboratory methods for controlling the baker's raw materials are not as complete as they should be.   In this respect, however, the baking industry is in company with many other industries and in the hands of a skilled man—and it must be emphasised that the cereal chemist must not only be skilled but of considerable experience—the results obtained from analytical methods are capable of safe interpretation.

F. E. T.
W. P. F.

CHAPTER 19

MILK

In the dairy industry today, the quality of milk and milk products is rigorously controlled by laboratory tests at all stages from the time the milk leaves the cow until the final milk or milk product in its container is delivered to the consumer. The tests used may be classified in three main groups:

Those used for routine control purposes in the industry.

More accurate tests used for legal purposes by public analysts and consultants.

Those used for research purposes.

This article is concerned mainly with the first group, but descriptions are given of some of those in the second group.

SAMPLING

For any laboratory test on any product the method of taking the sample is of the greatest importance. Not only must the sample be truly representative of the bulk, but special conditions sometimes have to be satisfied, e.g., the taking of samples under aseptic conditions for bacteriological tests.

In practice, milk is sampled for laboratory tests at the following points:

On the farm.

At the creamery or country collecting depot.

At the pasteurising or manufacturing depot.

During distribution to the public.

Sometimes identical tests are used at all four points of sampling, but at other times the method of sampling and testing may vary according to where the sample is taken.

On the farm, milk is taken from the churn direct into a bottle. At the country collecting depot the sample may be taken either from the churns on arrival, or from storage tanks after mixing. At processing and manufacturing depots the samples are almost invariably of the bulk milk, and, finally, during distribution the sample is normally one container, e.g., a one pint bottle. The complete testing scheme is summarised in Table I.

R

TABLE I

SYNOPSIS OF TESTS NORMALLY MADE ON MILK (1955)

Tests	Farmers' Milks at collecting depots	Bulk Milk at collecting depots and Pasteurising Dairies	Bottled Pasteurised milk at dairies and during distribution	Sterilised Milk
General and Physical 1. Sediment	+			
2. Temperature	(+)	+	(+)	
3. Cream line			+	
4. Freezing point	(+)	(+)	(+)	(+)
Chemical 1. Fat (Gerber)	+	+	+	+
2. Solids-not-fat (lactometer)	+	+	+	+
3. Turbidity				+
Biochemical 1. Phosphatase			+	
Bacteriological 1. Keeping Quality (smell, taste and C.O.B.)	+		+	+
2. Plate count	(+)		+	+
3. Coli	(+)		+	
4. Thermoduric count	(+)		(+)	
5. Methylene blue	+[1]	(+)	+	
6. Resazurin	+[2]	(+)		
7. Titratable acidity		+		
8. Thermophilic count				+
9. Platform (rejection) tests	+	+		
Special 1. Mastitis	(+)			
2. Tubercle bacilli	(+)	(+)		

Brackets indicate tests sometimes applied
[1] For designated producers   [2] For non-designated producers.

From the scientific point of view, tests on milk and milk products may be classified as general, physical, chemical, biochemical and bacteriological.

GENERAL TESTS

The best known general test for milk is that for sediment. During its passage from the cow to the dairy, various types of foreign matter can find entry into milk. These are mostly of no hygienic significance, but they are aesthetically objectionable, and consequently the amount of sediment in milk has to be kept to a minimum.

The Sediment Test

An operation of the simplest nature suffices for this purpose, and it consists merely of drawing or forcing a measured quantity of milk through a cotton filter, whereby any particles of dust, hair, chaff or the like are deposited upon the filter and their nature and amount may be assessed.

APPARATUS USED.—Various forms of apparatus are available, all of which are similar in principle. A well-known type of apparatus consists of a cylindrical body of cast aluminium alloy, provided with a hinged lid at the top and a cap at the bottom. The method of using this apparatus is as follows:

METHOD.—The cap is removed, together with the gauze disc which it carries. A special circular filter-pad of cotton cloth is now placed within the cap so as to lie flat upon the gauze disc (take care to have the nap side of the pad upwards). Finally, a circular rubber washer goes on top of the pad and the apparatus is ready for assembling, by joining the cap to the body. A half-turn of the bayonet-joint does this.

The milk under examination must now be thoroughly mixed. If contained in a bottle, this is readily accomplished by inverting the bottle a number of times, but if, as is more often the case, the milk is contained in a can or churn, it will be necessary to make use of a stirrer, or "plunger," to ensure thorough distribution of any solid matter which may be present.

All that now remains is to measure into the apparatus one pint of the mixed milk and, after securing the lid, to apply gentle pressure by means of the india-rubber bulb.

PRECAUTIONS.—It is well to note that, when the pressure is applied too vigorously, there is a tendency for the pad to become compressed and thus to retard the rate of flow very considerably. In cold weather, warming the milk to a temperature of about 100° F. will be found to facilitate the operation considerably.

EXAMINATION OF FILTER-PAD.—On removing the pad for inspection, we shall hope to find it quite free from any discoloration or deposit, thus indicating that the methods on the farm in question are satisfactory—from this particular point of view, at any rate. In some cases, however, we shall be disappointed. A dark circle in the centre of the pad will tell a tale of somebody's carelessness, of some precaution overlooked or neglected.

GRADED PADS FOR COMPARISON.—The depth of the discoloration will serve as a measure of the contamination, and where samples are examined systematically from a large number of farms it will be found useful to prepare a set of graded pads, so that the results of tests may be reported in well-defined terms. This system has the advantage that the receiver of the milk is enabled to keep a permanent record of the results obtained, and may yet return the actual pad to the producer with the object of demonstrating clearly the condition of the milk which he is sending.

VALUE OF THE TEST.—In spite of the very elementary nature of this test, its value should not be underestimated. Experience has proved that the visual evidence which it provides is readily appreciated by the cowmen and usually acts as an incentive to produce better results. Dirt can be a means by which large numbers of bacteria are introduced into milk, and due weight must be accorded to any test which contributes towards raising the standard of cleanliness, and thus safeguards health and improves keeping quality.

Other Tests

Other general tests are those of appearance, smell and taste. Any abnormal appearance indicates an abnormal condition, and so condemns the milk. The same applies to smell and taste. A sour smell indicates the growth of souring bacteria to an appreciable extent, a condition which is detected by the bacteriological tests described later. Souring can also be detected by taste. However, other conditions, sometimes physiological in nature, may be the cause of abnormal odours and tastes in milk. Certain types of feeding stuff and the eating of certain weeds by cows may result in the odoriferous substances passing through to the milk. Although objectionable, these flavours do not effect either the public health or the keeping quality of the milk, and gradually

pass off in time.   A red colour may be due to the presence of blood resulting from a ruptured blood vessel in the udder, or it may be due to a severe attack of mastitis.   Experience is necessary for the accurate diagnosis of abnormal smell, taste and appearance.

PHYSICAL TESTS

Temperature

One of the most important tests for milk is temperature. This is because the rate of growth of bacteria in milk, and consequently the rate of souring, is controlled almost entirely by the temperature.   Below 43° F. bacteria in milk do not grow; between 43° F. and 55° F. their growth is very slow, and above 55° F. they start to grow with increasing speed.   Milk held at 70° F. and above sours very quickly.   It follows, therefore, that one of the most important requirements in the dairy industry is to keep milk at about 40° F. or at a temperature as near this as possible during the whole of its history, i.e., from immediately after it leaves the cow until it is finally consumed.   For this reason the temperature of milk is a most important factor in the bacteriological test machinery, and is, in fact, one of the generally approved tests for bulk milk.   It is found in practice that the temperature at which bulk milk is received is a very reliable measure of its bacteriological condition.   The same reasoning applies to the temperature of milk in the churn as it arrives at the collecting depot from the farm, and also to the retail milk in the bottle as it is delivered to the housewife.   In practice, milk should be sent out from the retail dairy at a temperature between 40° F. and 45° F.

The Cream Line Test

The "cream line" or layer of cream, which forms at the top of milk when it is allowed to stand, is due to the fact that the specific gravity of milk fat is less than that of the watery part of the milk (skim milk).   The cream line is commonly considered by the housewife to be a measure of the richness of the milk, but this is a fallacy.   Broadly speaking, in raw milks the depth of cream line is proportionate to the fat content, but the ability of the fat globules to form a layer at the top is impaired by heating, so that over-pasteurisation can easily destroy the cream line of a

milk which is very rich in fat. Further, milk may be homogenised, i.e., the fat globules broken down to larger numbers of smaller globules. In milk which has been homogenised, the fat globules do not rise and so no cream line forms. Nevertheless, homogenised milk will have the same fat content as the milk before homogenisation and will be nearly twice as creamy in tea, coffee, etc., as unhomogenised milk. Sterilised milk is always homogenised, and so should never give a cream line.

The cream line of milk may be measured by a simple percentage given by the depth of cream line divided by the total height of the milk. If we now divide this percentage by the percentage of fat in the milk, we obtain the *cream line index* which is a measure of the cream line property of the milk independent of the fat content.

The Freezing Point Test

The fact that water can be added to milk without appreciably changing its appearance has made milk easily liable to adulteration. A most important aspect of the Food and Drugs law in this country is concerned with attempts to ensure that the public always receives pure unadulterated milk. In fact, the law requires that nothing must be added, and that nothing must be taken away from milk. Unfortunately, the high degree of variation in the composition of milk made it, until recently, difficult for the public analyst to detect adulteration. Fortunately, the freezing point test now provides him with a very accurate and reliable method for the detection of added water in milk.

Although the freezing point test is unquestionably the best for detecting adulteration, the test is essentially an empirical one, and it is most important to observe to the last detail the proper technique. Nearly all the tests which are now used for the routine control of milk are simple and can be easily learnt by the technician, but the freezing point and phosphatase tests require the most precise technique, and failure to observe all the precautions which are laid down in the official methods will easily lead to the obtaining of false results.

PRINCIPLE OF METHOD.—Milk does not have an absolute freezing point like water, and the test consists basically in lowering the temperature of the milk (super-cooling), to a definite point, and then "seeding" the super-cooled milk with a tiny crystal of ice. As the milk becomes solid, the temperature rises to a value

which is constant and reproduceable in any one sample of milk, provided that the technique is perfect.

The procedure which is now commonly used in this country is based on that first described by Hortvet, and so the test is sometimes called the "Hortvet test."

APPARATUS.—For a detailed description of the apparatus, reference may be made to standard text books.[3]   The apparatus consists essentially of an inner tube which contains the milk sample, a thermometer reading to $\frac{1}{100}$ ° C., and a thin metal rod for adding the crystal of ice.   This inner tube is held inside a slightly larger tube, the cavity being filled with alcohol to permit the conduction of heat.   These two tubes are held in a much larger vacuum flask which contains ether and a stirrer. The freezing of the milk is brought about by the production of cold resulting from the evaporation of ether, which is controlled by bubbling air through it.   When large numbers of samples are to be tested regularly, it is an advantage to have the special refrigeration unit freezing point equipment (Fig. 1).

CALIBRATION OF APPARATUS.—As the "freezing point temperature" is measured to $\frac{1}{1000}$ ° C. it is most important to calibrate

FIG. 1.—REFRIGERATION UNIT FREEZING POINT EQUIPMENT.

both the apparatus and the thermometer. This can most conveniently be carried out by obtaining a thermometer which has been calibrated in the National Physical Laboratory, and making a basic test with pure distilled water. The zero point of a very sensitive thermometer can vary from day to day according to the condition of the bulb. Having determined the zero point, the N.P.L. calibration chart can then be used to detect the slight inequalities which can occur in the stem of the thermometer. Finally, the apparatus and method can be checked by making freezing point tests on sucrose solutions from 7 to 8·75 per cent.

It is recommended that the novice should not make freezing point tests without a course of instruction. A detailed description of the method will be found in "Milk Testing."

INTERPRETATION OF RESULTS.—The freezing points of genuine milks from individual cows are found to vary from minus 0·530° C. to minus 0·560° C. When milk is adulterated, the freezing point depression diminishes, i.e., the freezing point approaches that of water. Public analysts and dairy chemists usually consider that milk has been definitely adulterated when the freezing point *depression* falls below 0·530° C. This allows a tolerance of about 3 per cent water, i.e., a farmer would only be liable to prosecution if the milk contained more than 3 per cent of water. With bulk milk such as the milk pasteurised and bottled in big dairies, the inequalities in individual cow samples are levelled out, and the unofficial standard adopted in the dairy industry today is a freezing point depression of 0·540° C., allowing a tolerance of 1 per cent added water.

CHEMICAL TESTS

Fat Content—Gerber Test

By means of this method, a batch of 16 samples may be examined for fat content in about 10 minutes and, with suitable precautions, the accuracy of the results will be within 0·1 per cent (plus or minus). The apparatus and reagents required are:

A quantity of test-bottles (butyrometers) of the Gerber flat-scale type;
A like number of lock-stoppers and a key;
Pipettes of 1 ml. and 10 ml. capacity respectively, preferably of an automatic type;

A bulb-pipette of 11 ml. capacity;
A rack to hold the Gerber bottles;
A centrifuge, driven by hand or power;
A fitted water-bath for immersion of the bottles;
Commercial sulphuric acid, specific gravity 1·820 to 1·825;
Amyl alcohol, free from furfural.

British Standard apparatus and methods are available and should always be used.[3]

BASIS OF TEST.—The test depends upon the fact that, when sulphuric acid and milk are mixed, heat is developed and the whole of the organic solid matter is dissolved, with the exception of the fat. The presence of a small quantity of amyl alcohol prevents charring of the fat and assists its separation under the action of centrifugal force.

METHOD.—For the operation of the test, a suitable number of the test bottles (usually 16) is placed, neck upwards, in a rack; 10 ml. of the sulphuric acid must now be introduced into each bottle and, for this purpose, it is most convenient to use an automatic pipette which delivers the required quantity with a single turn of the tap. If this piece of apparatus is not available, however, a bulb-pipette may be used, but, in this case, an additional safety bulb should be provided above the measuring bulb, in order that acid may not be accidentally drawn into the mouth.

The fact that cream rises more or less rapidly to the surface of milk when it is allowed to stand will obviously necessitate a thorough mixing of every sample before testing. It should become a matter of habit to effect a complete distribution of the cream throughout the body of the milk before removing a portion for analysis. As in the case of the sediment test, a metal stirrer with a long handle may be employed when the milk is contained in a can or churn. In general, however, the samples will be contained in bottles, having been transferred from bulk at some earlier stage. It is then only necessary to invert the sample bottle completely a number of times before opening, taking care that every particle of cream is dislodged from the inner surface of the glass and that thorough distribution is effected. This should be done *after* placing the acid in the Gerber bottles, the next stage of the test following immediately.

With a bulb pipette, measure 11 ml. of each of the various samples of milk into consecutive Gerber bottles. At this point, we do not wish the acid and the milk to mix and, by holding the point of the pipette against the side of the bottle and allowing the milk to flow slowly in an oblique manner, two distinct layers will be formed, milk above and acid below (Fig. 2).

An etched square on each of the Gerber bottles is provided for identification of the various samples by number, or otherwise, and, if use is not made of these, some definite system of rotation must
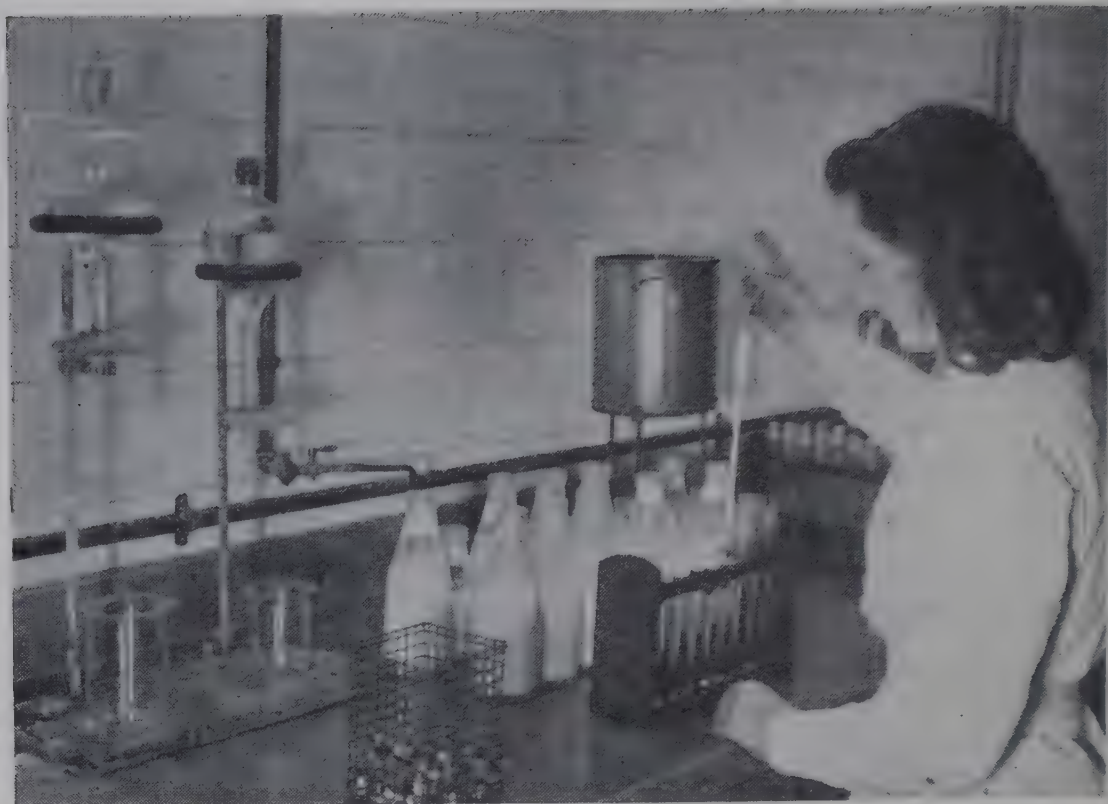
FIG. 2.—THE GERBER TEST FOR FAT. SECOND OPERATION.

be followed so that there is no possibility of confusion when it comes to recording the results.

One ml. of amyl alcohol is now added to each tube by means of a pipette similar to that used for the sulphuric acid.

Closure of the tubes is best effected by lock-stoppers, the metal rim of the stopper being held between the first and second fingers of the right hand, the thumb of which is then free for operating the key. By depressing the key, the rubber portion of the stopper is elongated and reduced in diameter so that it can be easily inserted in the neck of the bottle. On releasing the key, the stopper will revert to its normal shape and will then be found to be incapable of removal from the bottle by hand.

Everything is now ready for the shaking. Although metal covers for the rack can be obtained, by means of which it is possible to hold the bottles in position and shake the whole batch of 16 at one time, yet it is often found that analysts prefer to remove the bottles, four at a time, and shake them thus, two in each hand. In the course of the operation, considerable heat is developed, the contents of the tube assuming a dark coffee colour. The agitation should be continued until this colour is entirely uniform, no unmixed acid remaining.

Centrifuging must follow immediately, while the mixture is hot. Cylindrical metal cups are provided for the reception of the tubes, which are now placed therein, stopper first, of course. Here, again, we must take very particular note of the respective positions of the bottles so that we can subsequently remove them in the same order,

Any convenient system of marking may be employed. After attaching the lid of the centrifuge by means of the screwed boss in the centre, turn on the power and allow the machine to rotate for a full four minutes. This will cause complete separation of the fat from the acid solution of the other milk-solids. The acid will be thrown into the body of the tube while the fat, by reason of its lower specific gravity, will appear in the graduated neck.

On removal from the centrifuge, the bottles will have to be immersed for a few minutes in hot water to above the level of the fatty layer, and this water should be maintained between 60° C. and 70° C., as this is the temperature at which the bottles are calibrated to be read.

If the test has been properly performed, we shall now find a perfectly sharp line of division between the acid layer and the fat, and all that remains is to read off the percentage of the latter.

READINGS.—Taking the bottle, together with the stopper-key, in the right hand, as shown in Fig. 3, and viewing it against the light, we shall see that the appearance of the column of fat is similar to that illustrated in Fig. 4. The line dividing the fat from the acid will be found to be horizontal, whereas the top surface of the fat will be curved. Now, by light pressure with the thumb on the key, the horizontal dividing line may be slightly raised so as to coincide exactly with one of the *main* graduations, the numeral against that graduation being mentally noted. A rapid glance upwards will enable us to read, almost simultaneously, the level of the top of the fat column. In this case, it is the lowest point of the curved meniscus which is noted. A

FIG. 3.—GERBER TEST. FINAL OPERATION.

The line dividing the fat from the acid is horizontal; the level of the top of the fat column is taken at the lowest point of the curved meniscus. A simple subtraction will give the percentage of fat. In the above example the reading is 4·2 minus 1 equals 3·2 per cent.

KEY FOR STOPPER

FAT

MILK - SOLIDS (OTHER THAN FAT) DISSOLVED IN THE SULPHURIC ACID

ADJUSTABLE STOPPER

CC 60
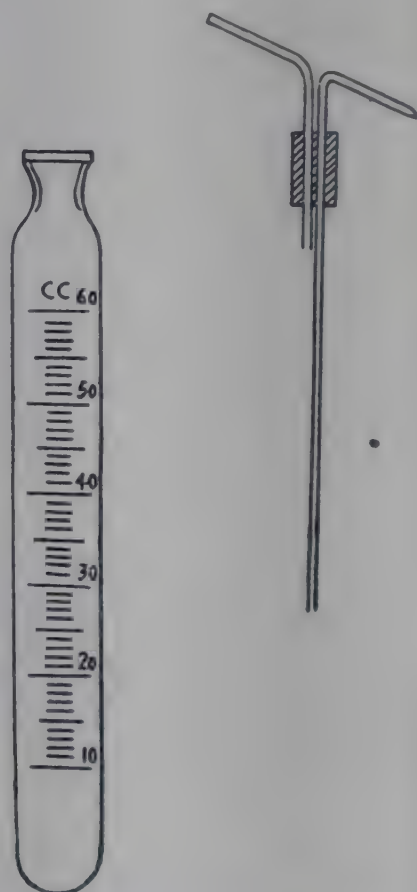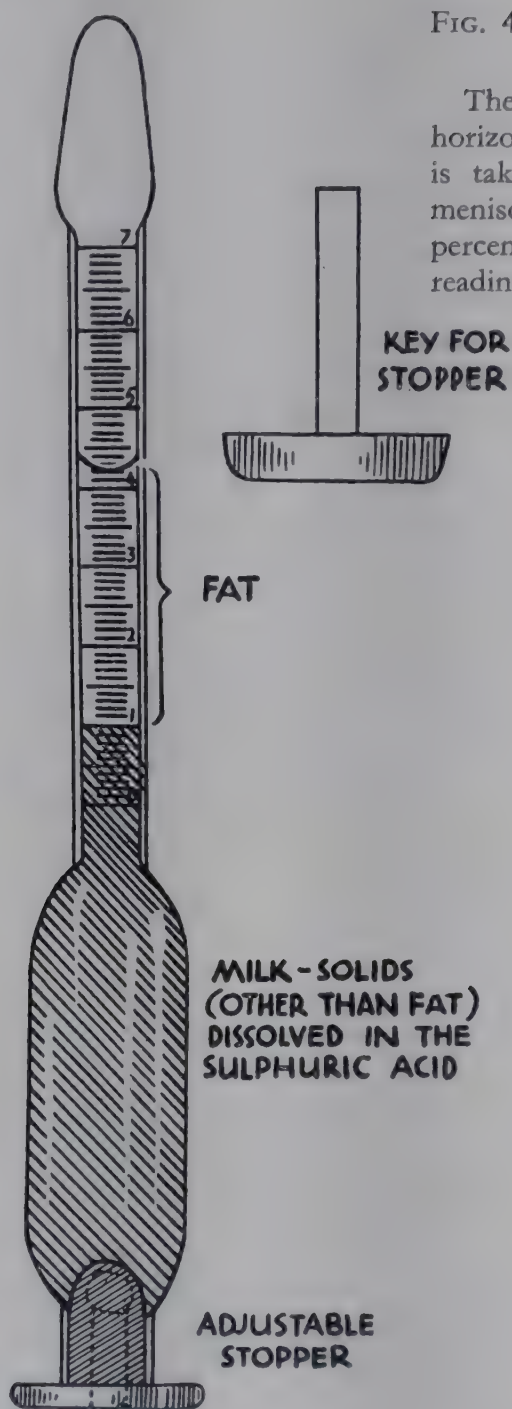
FIG. 5.—THE ROSE-GOTTLIEB TUBE.

simple subtraction will give us the percentage of fat; thus, in Fig. 4, the reading is 4·2 minus 1, equals 3·2 per cent. Although the smallest graduation is 0·1 per cent, it is customary to report results to the nearest 0·05 per cent, as read by eye.

A word concerning the *cleaning of the tubes* may not be amiss. If they are emptied while still warm and rinsed two or three times with hot water, they should be quite clean and fit for use again. Any deposit adhering obstinately to the glass must be regarded as a sure indication that the test itself has not been properly carried out.

The Gerber test is almost universally used in this country for

routine work.   For accurate determinations of fat, e.g., for legal purposes, a gravimetric extraction method must be used.   For this purpose the Röse-Gottlieb method is generally employed.

Fat Content—The Röse-Gottlieb Test

Only after making milk alkaline may the fat be directly extracted with ether.   A tube, graduated from 10 ml. to 60 ml., as shown in Fig. 5, is weighed, first empty, and then containing about 10 ml. of the well-mixed milk; 2 ml. of ammonia (sp. gr. about 0·9) is added and the tube is corked and shaken, with slight warming if necessary, until the contents present an entirely homogenous appearance and are free from flakes of protein.   The addition of 10 ml. of 95 per cent alcohol follows, with further shaking.   Now add 18 ml. of ethyl ether (0.720) and mix the contents of the tube thoroughly by repeated inversions.   Vigorous shaking may produce an emulsion, and should be avoided.   Finally, add 18 ml. of petroleum ether (b.pt. 40° C. to 60° C.) and mix again.

On standing, the formation of two layers will be seen, the upper being an ethereal solution of the fat, which may be transferred almost completely to a small weighed flask by means of wash-bottle tubes Two further treatments with small quantities of a mixture of equal parts of ethyl ether and petroleum ether will complete the extraction of the fat, the separated ethereal layer, in each case, being added to the main volume.

Alternatively, the Gottlieb tube may be centrifuged before removal of the main volume of the ether, and, in this case, separation of the ethereal and aqueous layers is more complete.   It is then sufficient to read the total volume of the former and to transfer an aliquot portion to the weighed flask, the fat in the *whole* volume being deduced by proportion.

All that remains is to evaporate off the ether by immersing the flask in hot water, with due precautions against fire, and then to heat it in the air-oven at 100° C. until constancy of weight is attained.   All the reagents used must be free from residue upon evaporation.

Solids-not-fat—Specific Gravity Method

This consists of an observation of the specific gravity of the milk, which can be used, as we shall show, as a measure of the total solid matter present and, indirectly, of the solids-not-fat.

ESTIMATION OF THE SOLIDS-NOT-FAT.—So far as specific gravity is concerned, milk may be regarded as a solution or suspension of its several ingredients in water, and each of these ingredients will exercise its own individual effect upon the net result.   Thus, the fat, being lighter than water, will tend to decrease the figure below unity, whereas the whole of the solids-not-fat are heavier than

water and will, in consequence, tend to raise the specific gravity. The composite result of these two opposite effects is to yield a figure approximating to 1·032 (at 60° F.) for normal milk.

FORMULA CONNECTING FAT, TOTAL SOLIDS AND SPECIFIC GRAVITY.—Raising or lowering the relative proportions of the various constituents will obviously result in an alteration of the specific gravity, and, after co-ordinating the results of tens of thousands of analyses, it has been found possible to evolve a formula connecting the fat, total solids, and specific gravity so that, after estimating two of these items, the third can be deduced mathematically. In practice, it is customary to estimate the percentage of fat and the specific gravity, and to infer therefrom the percentage of total solids. The figure for solids-not-fat then becomes available by subtraction.

SPECIFIC GRAVITY BOTTLE.—Where considerable accuracy is desired, the specific gravity may be determined by means of a 10 gm. or 50 gm. specific gravity bottle.

The bottle should be weighed: (a) empty, clean, and dry; (b) full of distilled water at 60° F.; (c) full of milk at 60° F.

The ratio $\dfrac{\text{weight of milk}}{\text{weight of water}}$ then gives the specific gravity at 60° F.

Solids-not-fat—The Lactometer

It is usually necessary to employ more rapid methods, and the use of a specialised form of hydrometer, known as a lactometer, is most common.

As usual, the milk must be thoroughly mixed before being tested. In observing specific gravities, however, it is essential that the mixing shall *not* be accomplished by vigorous shaking of the sample but by repeated inversion only. Violent agitation causes the milk to froth, and a large amount of air may become incorporated therein, a fallacious reading being obtained in consequence.

METHOD.—Pour a quantity of the milk gently into a glass jar of convenient shape and capacity, and insert the lactometer obliquely, holding it firmly near the top of the stem (Fig. 6). A quick but gentle twist between thumb and finger will serve to free the bulb from any adherent air bubbles. Now depress the instrument until the scale is completely immersed, then free it and allow it to find its own level.

FIG. 6.—USING THE LACTOMETER.

In taking the reading it is customary to omit the digits immediately before and after the decimal point and refer to a specific gravity of, say, 1·0325 as one of 32·5 degrees, and so on.   Although the scale is divided only into half-degrees, we shall find it quite possible to estimate to the nearest $\frac{1}{10}$ degree by eye.   Let us look closely at our lactometer. We shall see that the jar and its contents present an appearance similar to that shown in Fig. 7.   That is to say, the surface of the milk is

FIG. 7.—

APPEARANCE OF LACTOMETER
FLOATING IN MILK.
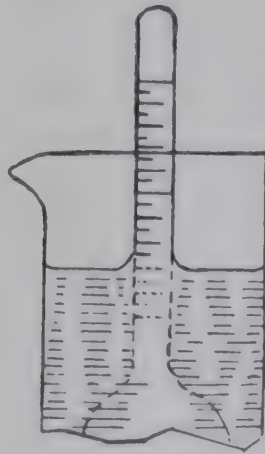
drawn up around the stem of the lactometer so that the actual point at which its horizontal plane would meet the stem is not visible.

Lactometers should be made to read *either* at the top of the meniscus *or* at milk level.   They should always be calibrated by a comparison of determinations on the same sample of milk by lactometer and gravity

bottle. The amount of the correction, once established, must be added to every observed reading. It is usually about one-half degree.

As in all estimations of specific gravity, the temperature will have to be taken at the same time and a suitable correction applied so as to report the result at 60° F. This is most easily done by means of the Richmond slide-rule.

In the upper left-hand corner will be seen two series of figures bearing the words "temperature" and "specific gravity" respectively, and it is only necessary to bring the observed gravity reading on the latter against the figure "60" on the former. The corrected gravity at 60° F. will then appear opposite the number (on the "temperature" scale) corresponding to the actual temperature at which the test was made.

Total Solids—By Calculation

The knowledge of the fat content and the specific gravity of the milk, which we have now gained, will enable us to measure the percentage of total solids by the application of a simple formula, which is as follows:

$T = 0.25 \, G + 1.2 \, F + 0.14$, where T represents the percentage of total solids.

G represents the specific gravity (in degrees).

F represents the percentage of fat.

In practice, it is more customary to make use of the Richmond slide-rule again. Examination of this will show that the upper right-hand corner of the slide bears an arrow which can be brought against any desired figure of the scale marked "Fat," and we shall now place this arrow so as to coincide with the percentage of fat which we have ascertained in the Gerber test. The lower edge of the slide is graduated from 20 to 36 in tenths and on this scale we must find the appropriate marking for the specific gravity of the sample in question. Opposite this marking, i.e., on the scale marked "Total Solids," we shall now be able to read off the actual percentage of total solids in the milk which we are examining. Subtraction of the fat from the total solids will give us the value for solids-not-fat.

The lacometer method for solids-not-fat, is, like the Gerber test for fat, a simple method for routine control, but for legal and other accurate purposes a gravimetric method must be used.

Total Solids—Gravimetric Estimation

A weighed quantity of the milk is dried, and the weight of the residual dry matter ascertained. For the purpose, flat-bottomed dishes about 7 cm. in diameter are required, and those made of stainless steel will be found suitable.

METHOD.—A dish, clean and dry, is covered by a watch-glass of slightly larger diameter, and weighed on an analytical balance. About 5 ml. of the well-mixed milk is then introduced by means of a pipette, and the dish is immediately re-covered by the watch-glass and weighed again. The purpose of the watch-glass is, of course, to prevent evaporation during this weighing, and it may now be laid aside.

By means of gentle rotation, spread the milk over the entire surface of the bottom of the dish and transfer the latter to the ring of a water-bath, allowing evaporation to continue for about half an hour after the milk-solids have apparently become dry. The prolonged heating at this stage reduces to a minimum the tendency for the solids to become brown subsequently, and enables constancy of weight to be attained more rapidly.

Now place the dish in an air-oven, the temperature of which is maintained between 100° C. and 102° C. The electrical form, with thermostatic control, is very convenient for the purpose. At the end of one hour remove the dish to a desiccator and allow to cool completely before weighing again (with the watch-glass, of course).

To ensure constancy of weight, the dish must be replaced in the air-oven for successive periods of 30 minutes, with intermediate weighings, until two consecutive weighings differ by less than one milligram. If the details of the method are carefully observed, this end is usually attained in two or three hours, and the weight of the dry matter is expressed as a percentage of that of the original milk, and reported as "total solids."

When an electrical air-oven is not available, a steam oven may be used but, in this case, a considerably longer time will be required to obtain constancy of weight.

The Turbidity Test (Applied to Sterilised Milk)

In October 1949 new regulations came into force for milk, and for the first time sterilised milk was recognised as a designated milk. The official test laid down was the turbidity test which is based on the fact that heating milk to at least the boiling point for a few minutes precipitates all the albumin. When milk is sterilised, it is usually held at temperatures in the range 220° F. to 240° F. for from 20 to 40 minutes. This heat treatment is much more severe than the minimum requirement to make a milk pass the turbidity test. The test therefore really only separates the strongly heated milks from the pasteurised milks. It cannot be assumed that because a "sterilised milk" passes a turbidity test that the milk is therefore sterile. In practice, usually from 25 to 75 per cent of all bottles of sterilised milk are really sterile.

S

Apparatus and Methods

A supply of conical flasks, 50 ml. capacity.

A supply of graduated cylinders, 25 ml. capacity.

A supply of test tubes conforming to British Standard Specification No. 625 (1935), 127/12.

A supply of filter funnels, 6 cm. diameter.

Two beakers, 400 ml. capacity.

A supply of Whatman folded filter papers, 12·5 cm. No. 12.

Weigh $4 \pm 0·1$ gm. of ammonium sulphate into a 50 ml. conical flask. Measure out $20 \pm 0·5$ ml. of the milk sample, and pour into the conical flask. Ensure that the ammonium sulphate dissolves by shaking for 1 minute. Leave for not less than 5 minutes and then filter through a folded paper into a test tube. When not less than 5 ml. of a clear filtrate has collected, place the tube in a beaker of water, which is kept boiling, and keep it therein for 5 minutes. Transfer the tube to a beaker of cold water, and when the tube is cool, examine the contents for turbidity by moving the tube in front of an electric light shaded from the eyes of the observer.

Interpretation

The test result is satisfactory if the filtrate shows no sign of turbidity.

Ash

The ash or mineral content of milk can easily be determined by first carefully drying a sample of milk and then heating it to a dull red heat. The ash consists principally of the phosphates and chlorides of calcium, potassium, sodium and magnesium. The ash content is not a figure of great importance today. Mastitis, or inflammation of the udder will result in an increase in the sodium chloride and a decrease in the lactose or sugar content of milk, and neutralisation of developed acidity by the addition of lime or soda will both increase the ash content.

Method.—The method involves the controlled heating of milk after preliminary drying. Heating is normally carried out until a constant weight of ash is obtained. Owing to the fact that a certain amount of chloride may be volatilised if heating takes place at a temperature higher than 550° C., it is recommended that for accurate work a thermostatically controlled muffle furnace should be used. Weigh accurately about 10 gm. of milk in a platinum, porcelain, silica or other suitable dish which has been ignited, cooled in a desiccator charged with efficient desiccant and weighed. Evaporate to dryness and ignite in a muffle furnace at a temperature not exceeding 550° C. until the ash is free from carbon. Cool in a desiccator, weigh, and calculate the percentage of ash.

BIOCHEMICAL TESTS

There is only one biochemical test which is used for the routine control of milk, and that is the phosphatase test. It is applied only to pasteurised milk and depends upon the fact that the enzyme phosphatase, which occurs naturally in milk, is destroyed by heat treatment slightly in excess of that necessary to kill the tubercle bacillus, which is the most heat resistant disease organism normally found in milk.

The Phosphatase Test

Pasteurised milk was first recognised as a designated milk in 1923, and for many years the bacteriological standard was a plate or colony count of 100,000 per ml. Pasteurisation usually destroys from 90 to 99 per cent of all the bacteria in raw milk and, although bacteria are naturally growing continuously during the handling and distribution of milk, it was considered that any efficient dairyman should have no difficulty in delivering to his customers a pasteurised, bottled milk containing fewer than 100,000 organisms per ml. However, in course of time it was realised that the proportion of thermoduric (i.e., surviving pasteurisation) organisms in raw milk could become very high, especially if the milk producer allowed his utensils and equipment to get into an unhygienic condition. The presence of milk resides, especially in milking machines, and subsequent half-hearted cleaning and sterilisation could lead to very high counts of these thermoduric bacteria in the raw milk, so that no pasteurisation process, no matter how efficient it might be, could reduce the total count below this basic number of thermoduric bacteria in the raw milk directly attributable to bad conditions of production. For this reason, the colony count standard for pasteurised milk fell into disfavour, and in 1946 it was officially replaced by the phosphatase test.

Principle of Method.—Phosphatases are enzymes like pepsin and invertase, which have the power to bring about certain chemical reactions, usually the splitting of certain complex substances to more simple compounds. Phosphatases have the power to split off inorganic phosphate from the compounds of phosphoric acid with organic substances. In the official phosphatase test a compound of phenol and phosphoric acid is split

into free phosphoric acid and free phenol. Either of these compounds can be estimated, but the official test estimates the phenol which is produced. As only very minute quantities are involved, a special colorimetric micro-method has to be used and the resultant blue colour is normally measured in a Tintometer all-purposes comparator using a special disc, the various colours of which correspond to different amounts of free phenol.

METHOD.—As with the freezing point test the technique for the phosphatase test must be carried out with the most rigorous attention to detail. A full description of the method and apparatus will be found in "Milk Testing," but the method is basically as follows:

To 10 ml. of the buffer-substrate solution, which contains disodium phenyl phosphate as a substrate, and sodium veronal as a buffer, is added 0·5 ml. of the well mixed sample of milk. Three drops of chloroform are added, the contents of the tube well mixed and incubated at 37° C. for 24 hours. The solution is then cooled, and 4·5 ml. of the test reagent (Folin and Ciocalteu's phenol reagent) added, the solutions mixed, allowed to stand for 3 minutes and then filtered into a test-tube marked at 10 ml. Two ml. of sodium carbonate solution are then added to the 10 ml. of filtrate, the solution mixed and the test-tube placed in a bath of boiling water for exactly 2 minutes. The tube is then cooled, and the intensity of blue colour measured, using the Tintometer disc. Using this official technique, a value of more than 2·3 Lovibond blue units indicates that the milk has not been efficiently pasteurised, i.e., some phosphatase has survived the pasteurising process, or that some raw milk is present.

PRECAUTIONS.—It is most important that all the precautions laid down shall be properly observed as it is very easy to get a false positive result. In particular, a test made with the reagents only must have a value not exceeding 0·5 L.B.U., and a test made with milk, but without the 24 hour incubation period, must give a value not exceeding 1·5 L.B.U.

The phosphatase test can rightly be regarded as one of the most important tests ever devised for milk. Certainly of all the tests which have been suggested for the efficiency of pasteurisation, the phosphatase test is universally regarded as the best. The only disadvantage of the official method is the necessity for a very elaborate and careful technique.

Normally the test will detect the presence of 0·2 per cent of raw milk with certainty, and a slight lowering of the pasteurising temperature and or a shortening of the pasteurising time will also be revealed in the phosphatase test.

BACTERIOLOGICAL TESTS

The bacteriological testing of milk has always been a most controversial matter. The earliest laboratory tests were basically of a medical origin, and were, in fact, identical with those used for water, i.e., the plate count and coli tests. Excellent as these are for water, it was in time realised that these tests had their limitations for milk. For example, the plate count applied to milk was often of limited value because the organisms found in milk varied widely in their chemical activities, some producing rapid souring, and others having no effect at all. For this reason there was sometimes only a very poor correlation between the plate count and the keeping quality of a milk sample. Further, the plate count is a measure of the total living organisms in milk and does not differentiate between the dangerous disease organisms and the quite harmless saprophytic types. The coli test, which is probably the most valuable of all tests for water because certain types of coliform organisms in water are a reliable indication of recent faecal pollution, has a different significance for milk. Coliform organisms are obnoxious in milk because they produce rapid souring and taints, but many coliforms are of no public health significance and even the faecal types do not necessarily indicate recent faecal contamination of milk.

Quite naturally the public health authorities and the dairy bacteriologists in industry test milk for different purposes, and bacteriological tests can be classified quite sharply into those for disease organisms and those used to measure the commercial or keeping quality aspect of the milk. Unfortunately there is no simple reliable test for the presence of disease bacteria in milk. The most important is that for tubercle bacteria, and for reliable diagnosis it is necessary to inoculate the centrifugal deposit from a volume of milk into two guinea-pigs, the test taking up to six weeks. Microscopic examination is unreliable, although the tubercle bacteria, being acid-fast, can be made to take up a characteristic stain. For the specific tests for disease organisms textbooks on medical bacteriology, or *A Dictionary of Dairying* should be consulted.

The various bacteriological tests used in the dairy industry will now be described.

1. The Keeping Quality (Smell, Taste and Clot-on-Boiling (C.O.B.) Test)

Practical experience in the dairy industry over the last 50 years has served to emphasise the fact that none of the classical bacteriological tests is a very accurate measure of the keeping quality of milk from the consumer's point of view. The life of a milk is generally considered to be at an end when it clots on boiling, this usually occurring at a titratable acidity (expressed as total lactic acid) of 0·23 per cent. Milk is also considered to be unusable when it has developed a detectable "smell" or taste. By far the simplest and most accurate way of determining when milk has reached this condition is to make subjective tests for smell, taste and clot-on-boiling, and the modern trend is to lay more emphasis on this test from the point of view of determining the life of a milk from the distribution point of view.

These tests are very simple to make but they have the obvious draw-back that smell and taste are subjective qualities and cannot be measured objectively like the plate count and coli tests. Nevertheless it is probably that these 3 tests, commonly referred to as the *keeping quality test,* will assume more and more significance in the future.

2. The Plate Count or Colony Test

All bacteriological tests have to be performed under what are commonly called aseptic conditions and a bacteriological technique can only be acquired by a personally supervised training under an experienced bacteriologist. Detailed descriptions of this and subsequent bacteriological tests will be found in "Milk Testing"[3] and only the basic descriptions of the tests can be given here.

The plate or colony count of milk depends upon the fact that when milk is mixed with a nutrient solid medium, each bacterial cell or group of cells then proliferates at a very fast rate when the medium is held at a warm temperature (usually 30° C. or 37° C.). Although single bacterial cells are not visible to the unaided eye, when the cells have grown to the extent of millions they form a colony in the nutrient medium and this is easily seen without a microscope or even a lens. The colony count test is performed by mixing $\frac{1}{10}$, $\frac{1}{100}$ and $\frac{1}{1000}$ ml. of the milk with about 6 ml. quantities of the nutrient agar medium in plates (or Petri dishes)

and the plates are then held at 30° C. or 37° C. for 2 or 3 days. The bacterial colonies are then visible as small white or coloured particles. The dilutions scheme for plate and coli tests is shown in Fig. 8.
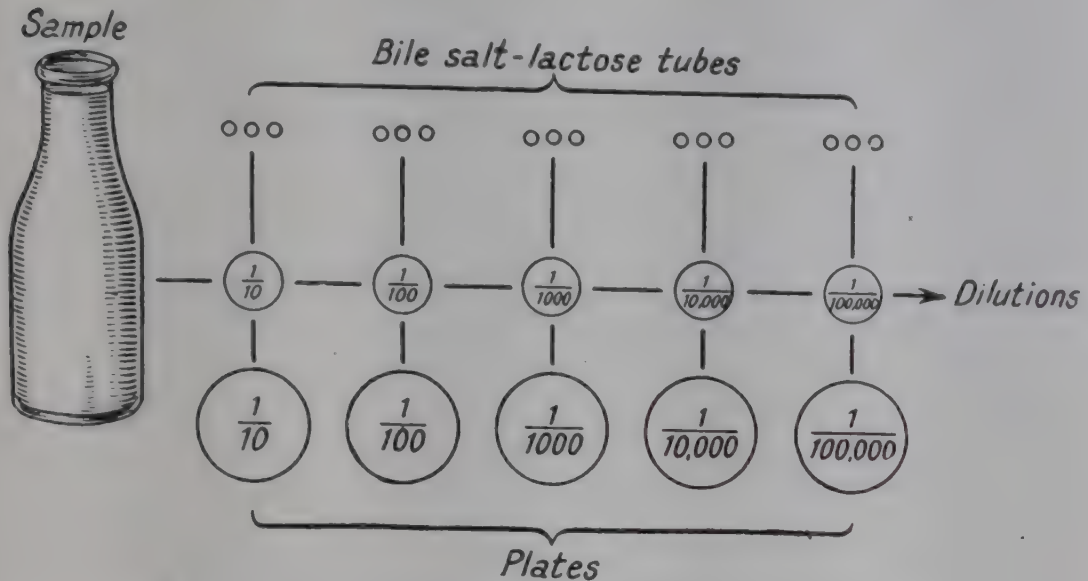
FIG. 8.—DILUTIONS SCHEME FOR PLATE AND COLI TESTS.

3. The Coli or Coliform Test

The coliform group consists of bacteria which may be derived from faecal contamination or from plants, but they all have one characteristic feature in common—they are able to ferment the lactose or sugar in milk with the production of acid and gas. The test for coliform organisms therefore consists in mixing various quantities of milk with a liquid nutrient broth which contains lactose and a colour indicator for acidity.

The broth is held in a test tube which contains an inverted small (Durham) tube. If the dilution of milk contains coli bacteria, they will grow and ferment lactose in the broth with the production of acid, which changes the colour of the indicator, and gas, which collects in the small (Durham) tube. By examining a series of tubes which contain various dilutions of milk and noting which of them have turned acid and produced gas, it is possible to estimate roughly the numbers of coliform organisms in the milk.

INTERPRETATION.—It must be emphasised that, unlike the test when applied to water, the presence of coli in milk does not necessarily indicate faecal (manurial) contamination. It does, however, indicate dirty methods in production and handling,

and especially failure to clean and sterilise the utensils and equipment which have been used for the production and transport of the milk.

DETECTION OF FAECAL COLI.—The method described which is based upon the production of acid and gas in MacConkey broth, detects both the true coli types of animal or faecal origin, and the aerogenes types which are derived from plants.

If it is desired to ascertain if the faecal type is present, a loopful of the broth from a positive tube (i.e., one showing acid and gas) is transferred to a fresh tube of MacConkey broth and the tube incubated at $44 \pm 0.5°$ C. The temperature control of the water bath must be within these limits. If acid and gas is then produced at this temperature, it indicates that the faecal type of coli is present.

4. The Laboratory Pasteurisation Test for Thermoduric Organisms

When milk of good bacteriological quality is pasteurised, only about 1 per cent of the total numbers present in the raw milk survive. These surviving organisms are thermoduric by definition. If a large proportion of the bacteria in the raw milk are thermoduric naturally a larger proportion will survive, and the bacterial count of the pasteurised milk may be high. If the pasteurising equipment, pipe lines, filler and bottles are nearly sterile, as they should be in a good dairy, there will be very little post-pasteurisation contamination of the milk. If, however, the equipment is in an unhygienic condition, the bacterial count of the pasteurised milk will be greatly increased, and these contaminating bacteria will be largely killed by a re-pasteurisation of the milk. It is possible, therefore, by laboratory re-pasteurisation of pasteurised milk to assess the hygienic condition of the dairy plant. Thus a high count on pasteurised milk which is markedly reduced by laboratory re-pasteurisation indicates an insanitary plant, whereas, a high count which is very little reduced by laboratory re-pasteurisation, indicates that the original flora of the raw milk was largely thermoduric.

5. Direct Microscopic Count

It is possible to count the total number of bacterial cells, dead and alive, in milk by spreading 0·01 ml. of milk over an area of 1 sq. cm., drying the film carefully, staining it with Newman's

stain, and examining it under a microscope, using a $\frac{1}{12}$ in. objective with oil immersion. This method is popular in the United States but is hardly ever used in the United Kingdom. It has the advantage that it gives the result in a matter of minutes, and various types of micro-organisms can be distinguished. However, it cannot differentiate living from dead organisms, and is of little use for good quality milk, e.g., for milk with a count of less than 500,000. Nevertheless the test could be used to a greater extent in this country, especially where a quick result is desired. On the average, the direct microscopic count gives a value about four times that given by the plate or colony count for raw milk, and about eighty times for pasteurised milk.

6. Dye-reduction (Methylene blue and Resazurin) Tests

During the last 20 years the classical plate count and coli tests have been replaced almost entirely by dye-reduction tests. The dyes normally used are methylene blue and resazurin. These tests measure not the numbers of bacteria, but give an overall measure of the chemical activity, and especially the respiration of all the organisms in the milk. At a certain point in the growth of bacteria oxygen is removed from the dye, and this chemical change alters its colour. Thus methylene blue will become colourless, and resazurin will change from blue through purple to pink and ultimately white. The speed with which this colour change takes place is a fairly accurate measure of the numbers of bacteria in the milk.

The test differs from the colony count in that it gives a considerable "weighting" to the chemical activity or intensity of metabolism of the bacteria. Thus all living bacteria may form colonies on the agar medium, but some will have a very drastic effect on methylene blue or resazurin, while others have little or no effect. The types which are active against these dyes are the coliforms, streptococci and staphylococci, and these are, broadly, the types which sour milk and reduce its keeping quality. Dye-reduction tests are therefore fundamentally sound. Normally they are made at 37° C. in order to get a quick result. Thus, a milk which reduces methylene blue in $4\frac{1}{2}$ hours in summer will fail the standard for Tuberculin Tested milk. If the test is made at room temperature, say 18° C. it will take about three

times as long but the result will correlate better with the commercial keeping quality of the milk.

METHOD.—The technique is the same for both the methylene blue and the resazurin tests. Ten ml. quantities of the milk samples are placed in sterile test tubes, 1 ml. of a standard dye solution added, the contents mixed and the tubes placed in a water bath, usually at 37° C. The tubes, which are closed with a rubber bung, are examined at half hourly intervals. If no change has taken place, the tube is inverted, reverted and replaced. If the methylene blue is reduced (i.e., the blue colour has disappeared) the test is finished. With resazurin, the tubes are placed in a special comparator with a resazurin disc and the colour tint matched, the result being recorded as a disc number which may vary from 6 (the initial value) down to 0. Very roughly, the resazurin disc number at the end of 1 hour is the same as the time in hours required to reduce methylene blue.

INTERPRETATION.

1. *Raw designated milk.* Tuberculin-Tested milk (and formerly Accredited milk) must not reduce methylene blue in $4\frac{1}{2}$ hours from the 1st May to the 31st October, and in $5\frac{1}{2}$ hours from the 1st November to the 30th April.

2. *Pasteurised milk* is held overnight at room temperature and the next morning must not reduce methylene blue in half-an-hour. This standard anticipates the clot-on-boiling stage by a few hours and so is a safeguard that the milk is of usable quality on the morning after delivery.

3. *Ordinary raw milks.* Under the former National Milk Testing Scheme, and now under the Joint Milk Quality Control Scheme, ordinary or undesignated farm milks are tested on arrival at the collecting depot by the 1 hour resazurin test. The standards are: Disc 6 to 4—satisfactory; $3\frac{1}{2}$ to 1—doubtful; $\frac{1}{2}$ to 0—unsatisfactory.

For further technical details and official aspects see "Milk Testing."[3]

Platform or Rejection Tests

One of the most important problems of the laboratory in the modern dairy is to safeguard the quality of the final product, and this can only be achieved by the systematic testing of the raw material and rejection of unsatisfactory milk supplies. Both farmers' herd bulks in churns or cans, and bulk milk in tankers should be regularly tested for bacteriological quality. Whereas

a milk which is low in fat or solids-not-fat may be satisfactorily blended with milk which is high in fat and solids-not-fat, milk of bad bacteriological quality cannot be similarly blended with good milk because a very small proportion, as little as 0·1 per cent, of a high count milk may turn a bulk of low count milk sour. Under the most favourable conditions one bacterial cell can become two in about $\frac{1}{4}$ hour, which means that one cell can become a million in 5 hours. For this reason it is essential to apply at least a very simple "rejection test" to every batch of milk which is received at either the country collecting depot or the town pasteurising dairy. Tests which are commonly used are smell, titratable acidity, ten-minute resazurin and temperature. These will now be discussed.

1. SMELL.—This is the most sensitive test for off-flavour in any food, and the sense of smell can often detect obnoxious substances in concentrations far below those at which the most delicate laboratory tests begin to operate. The minimum test, therefore, which should be applied to any can or tanker of milk is smell, and a trained "checker" or platform operative is a most important member of the dairy staff. Naturally it requires experience and skill to differentiate the smell resulting from bacterial growth from those due to the nature of milk itself and the effect of feeds and weeds.

2. TITRATABLE ACIDITY.—Fresh milk contains practically no lactic acid but as bacteria begin to grow, lactic acid is produced by the fermentation of the lactose or milk sugar. When fresh milk is titrated with caustic soda using phenolphthalein as an indicator, an appreciable value, about 0·14 per cent "lactic acid" is obtained, and only titration values in excess of this figure are due to the lactic acid proper which has been produced by souring. Unfortunately this initial value, which is due to proteins and phosphates in the milk, is rather variable, and the acidity test is not a reliable measure of souring for herd consignments, and still less reliable for a can of milk. For bulk milks which may consist of 1,000 gals. upwards, the test is a reliable measure of souring and is one of the recognised tests for this purpose.

3. TEN-MINUTE RESAZURIN TEST.—It has been mentioned that resazurin reacts more quickly to bacterial action than methylene blue, and milks of very high count, e.g., over 10

million per ml., usually produce some change in the colour of resazurin in 10 minutes at 37° C. This test is now generally regarded as the most reliable platform or rejection test for milk, and whenever a can of milk is found to have a suspicious odour a ten-minute resazurin test should be made. This test is also one of the recognised tests for bulk milk.

4. TEMPERATURE.—There is usually a good correlation between the temperature of a farm or tanker bulk milk on arrival at the buyer's premises, and its bacteriological quality. The correlation is not so good for farm milks, as a milk of good keeping quality can be produced by sterile equipment and with no cooling, and conversely, a dirty equipment may give a milk of bad bacteriological quality even though it is cooled. For bulk milk however, the correlation is usually very good, and for this reason temperature is one of the three recognised tests for bulk milk.

Standards

There are no legal standards for the bacteriological quality of milk on acceptance, but the following standards which were laid down under the National Milk Testing Scheme are still generally observed today.

CRITERIA FOR REJECTION

Test	Farmers' milks in cans	Bulk milk in tankers
Smell	Taint	Taint
Titratable acidity	—	Greater than 0·17% lactic acid
Ten-minute resazurin	Less than disc 4	Less than disc 3
Temperature	—	Greater than 43° F.

Mastitis

Mastitis is a disease of the udder which results in the secretion of milk poorer in lactose and casein and higher in salt content. Mastitis milk is always more alkaline than ordinary milk, and for these reasons can be the cause of inferior manufactured products. Fortunately, the causative organism of mastitis is of

no public health significance, but for special purposes control tests for mastitis may be necessary. The simplest tests for routine purposes are:

1. A cell count, the technique for this being identical with that for the microscopic count described above.

2. A catalase test which is an indirect way of measuring the cells.

3. The chloride test, which depends upon the fact that mastitis increases the chloride content of milk, a figure of greater than 0.12 per cent being indicative of serious mastitis.

Thermophilic Count

Thermophilic bacteria are those able to grow at holder pasteurising temperature (145° F.), and it is sometimes desirable to measure the numbers in milk. The technique for this is identical with that for the plate count described above, but the plates must be incubated at 145° F. An incubation time of 24 hours is usually sufficient, and the Petri dishes should be sealed with plasticine or a jar of water placed in the incubator to minimise the evaporation of water from the agar medium. Thermophilic organisms are not important today when the high temperature-short time method of pasteurisation is almost universal, but formerly, when the classical holder method was commonly employed, these organisms often led to serious economic faults.

J. G. D.

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* Advanced

MEAT PRODUCTS

Meat Extract

1. SAMPLING.—Those extracts that are not too tenacious should be thoroughly mixed, using a stainless steel rod, in the original container or the total contents may be removed and then mixed.

Some extracts may require to be heated somewhat before mixing is possible. This heating is best done in the original container before opening, and should be of as short a duration as is compatible with rendering the extract soft enough to mix thoroughly. The heating takes place in a 100° C. oven.

2. MOISTURE.—Weigh 2 gm. of the material into a 50 ml. beaker (Fig. 1) and dissolve in 20 ml. of hot water (Fig. 2), allowing to remain on a steam bath with stirring until solution is complete.

Transfer to a dry tared nickel basin containing 25 gm. of dry sand. Mix thoroughly (Fig. 3) and evaporate off the water on the steam bath (Fig. 4).

Place in the oven (Fig. 5) at 100° C. for 16 hours, cool in a desiccator (Fig. 6) and weigh. The increase in weight is due to the solids contained in the 2 gm. taken for determination.

3. ASH.—Weigh into a tared crucible 2 gm. of the extract. Heat the crucible and contents under an infra-red lamp until well charred (Fig. 7).

Heat the crucible cautiously over a bunsen burner until the residual matter ceases to emit smoke on fairly strong heating. Cool, and transfer to a cold muffle furnace (Fig. 8). Muffle at not more than 550° C. until consecutive weighings are constant.

4. CHLORIDE as SODIUM CHLORIDE.—Transfer the ash from the previous determination to a 150 ml. beaker using a jet of hot water. Add 10 ml. of dilute nitric acid (1 in 5), stir and then add from a pipette 25 ml. N/10 silver nitrate. Stir and heat on a steam bath until the precipitated silver chloride is well coagulated. Cool.

Filter through a Whatman No. 1 paper and well wash the precipitate and paper with cold water. To the filtrate add 5 ml. of ammonium sulphate indicator* and titrate the excess silver nitrate with N/10 ammonium thiocyanate until a slight red colour persists.

1 ml. N/10 silver nitrate ≡ 0·005846 gm. NaCl.

* 10 per cent in nitric acid (1 + 9).
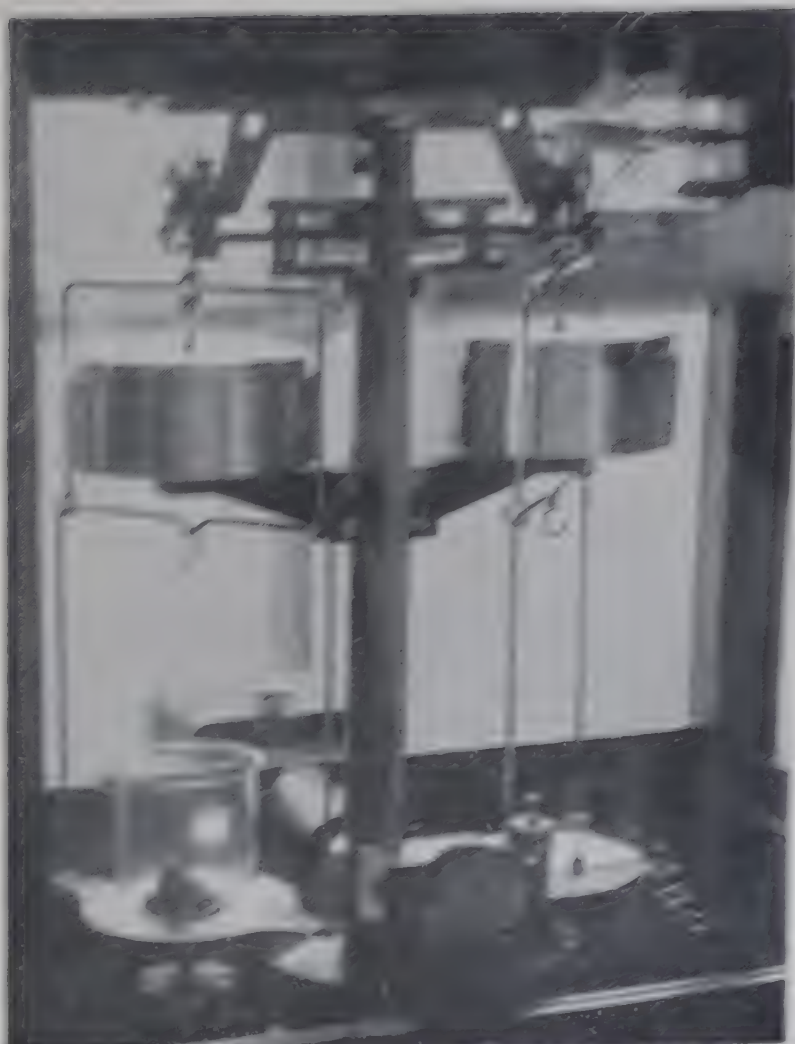
Fig. 3.—Moisture
Determination.

Mix sample with
ignited sand in a
nickel dish.

Fig. 4.—Moisture Determination.   Place sand basin on a boiling water bath.

FIG. 5.—MOISTURE DETERMINATION.

Transfer sand basin to electric oven at 1,000° C.
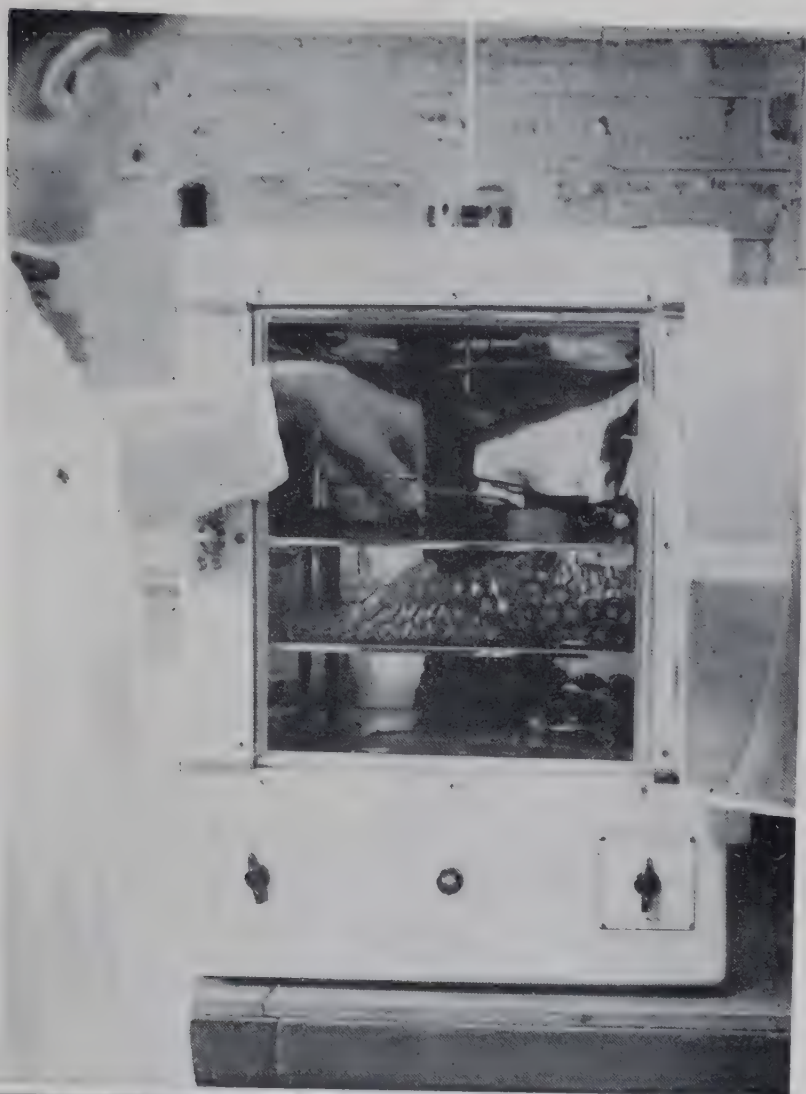
FIG. 6.—MOISTURE DETERMINATION.

Transfer sand basin to a desiccator.

T
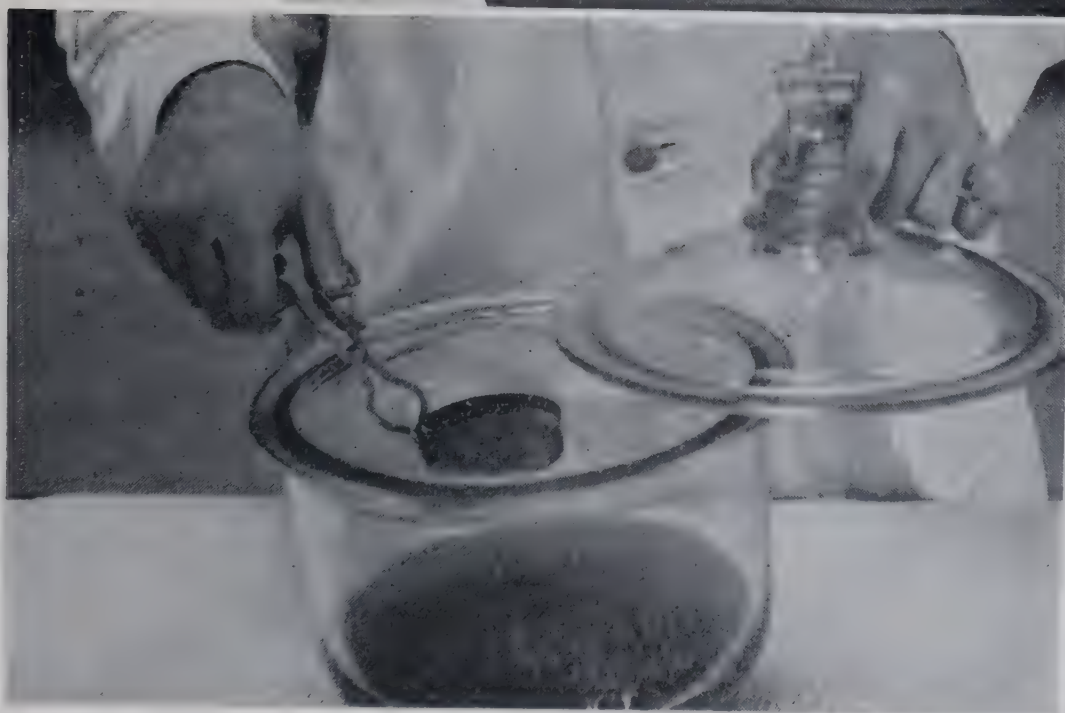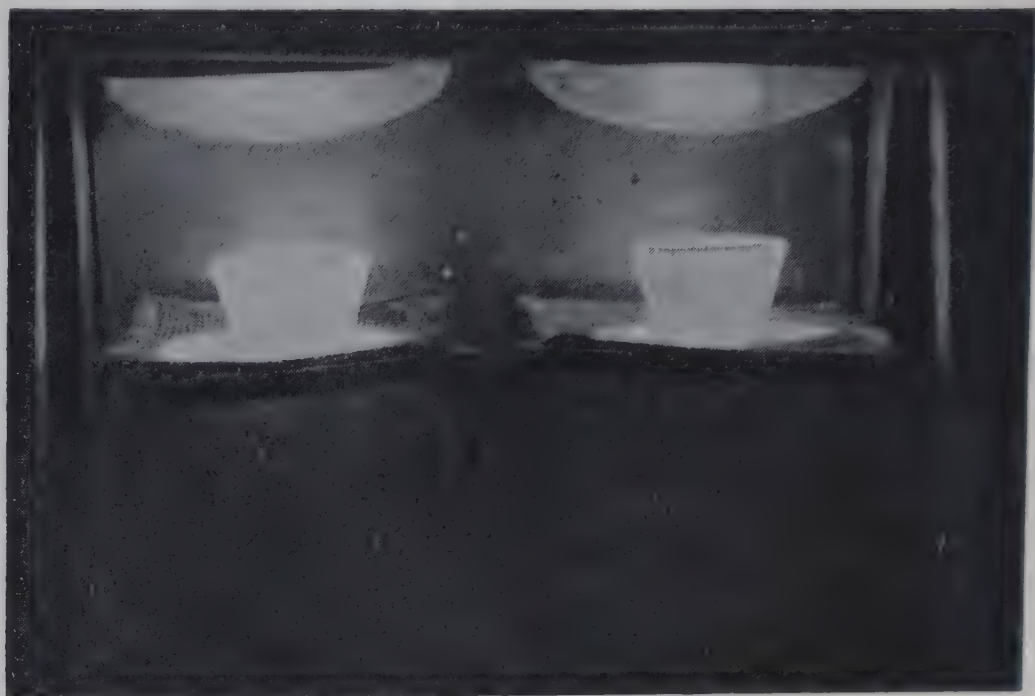
FIG. 7.—ASH DETERMINATION.
Heat sample under infra-red lamps prior to igniting.
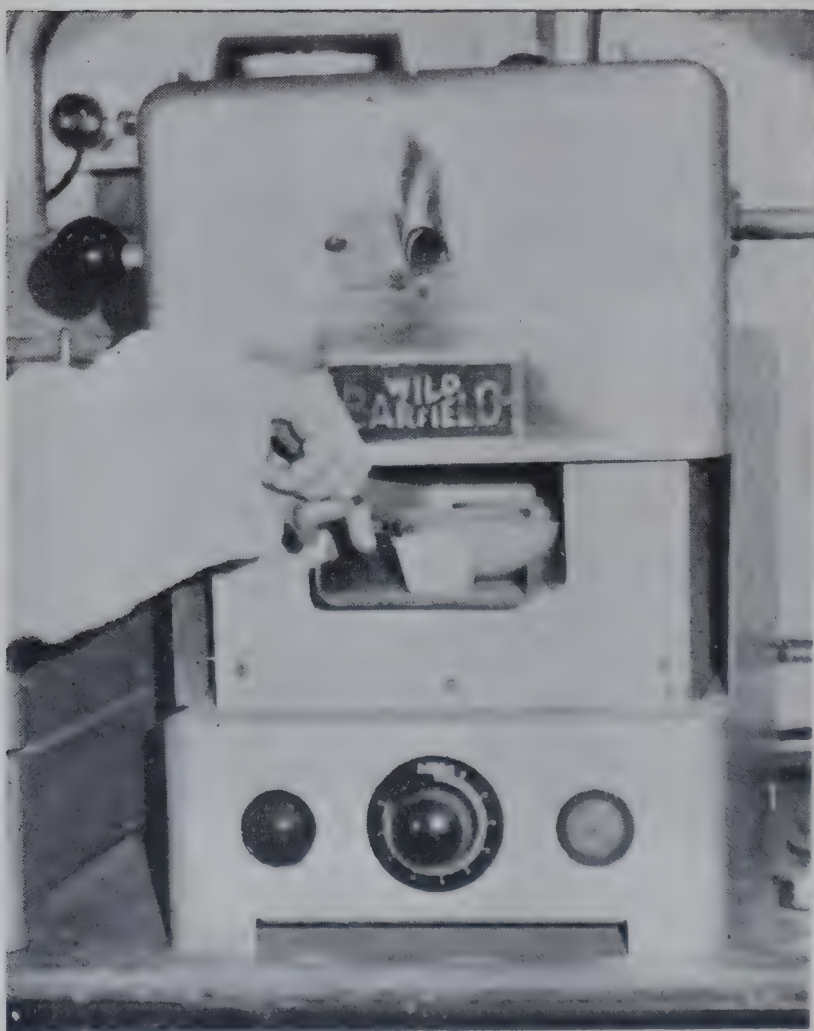
FIG. 8.—
ASH DETER-
MINATION.

Muffle at not
more than
550° C. until
consecutive
weighings are
constant.

5. TOTAL NITROGEN.—Weigh 1 gm. of extract into a small beaker, and transfer to a 500 ml. Kjeldahl flask after effecting solution in water.

Add 1 ml. of 20 per cent copper sulphate solution, 25 ml. of sulphuric acid, and 10 gm. of anhydrous sodium sulphate.

Mix, warm cautiously over a bunsen flame in a fume chamber, until fumes of sulphuric acid are evolved. Place a small Pyrex funnel in the neck of the flask and continue the heating more strongly so that the acid boils smoothly (Fig. 9).

Continue this heating until 3 hours from the time of clearing.

Cool.

After cooling dilute the contents of the flask with 100 ml. of water.

Connect to an all glass Kjeldahl distillation apparatus (Fig. 10). Distil into 25 ml. of N/2 sulphuric acid, using 80 ml. of 50 per cent NaOH solution to effect release of all the ammonia.

Titrate with N/2 sodium hydroxide using methyl red/methylene blue as indicator.

$$1 \text{ ml. N/2 sulphuric acid} \equiv 0.007 \text{ gm. Nitrogen}$$
$$\equiv 0.04375 \text{ gm. Protein}$$

Solution of Methyl Red and Methylene Blue.

Mix 200 ml. of a 0.05 per cent w/v solution of methyl red in alcohol (20 per cent) with 4 ml. of a 2 per cent w/v solution of methylene blue in water.

Fig. 9 shows three Kjeldahl flasks, with Pyrex funnels inserted, arranged for heating in a fume chamber.
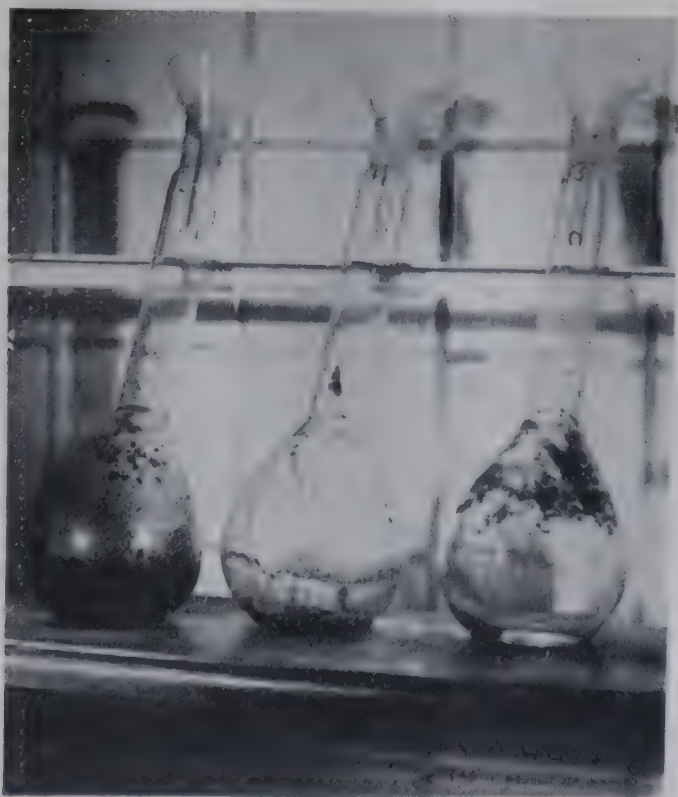
FIG. 9.—A KJELDAHL DIGESTION APPARATUS FOR TOTAL NITROGEN DETERMINATION
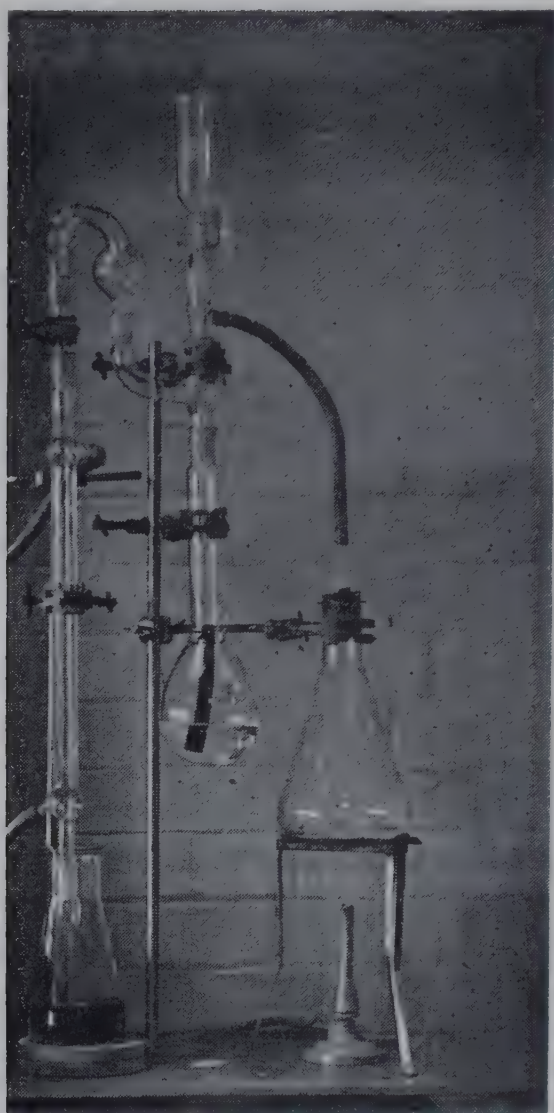
Fig. 10.—
A Distillation
Apparatus for
Total Nitrogen
Determination.

6. Total Creatine and Creatinine as Creatinine.—Solutions required:

Hydrochloric acid—A 2N solution.

Sodium hydroxide—A 2N solution.

Picric acid—A 1 per cent solution.

Stock creatinine zinc chloride solution—1·603 gm. of pure crystalline creatinine zinc chloride made up to 1000 ml. with N/10 hydrochloric acid. This solution is stable for at least six months, and an aliquot should be diluted ten times, immediately prior to use, so that 1 ml. ≡ 0·1 mg. of creatinine.

Hydrolysis of Extracts.

Weigh into a small flask beaker 1 gm. of extract and dissolve in 10 ml. of hot water. Add 10 ml. 2N hydrochloric acid and insert small funnel in neck of flask. Heat under reflux in a boiling water bath for 2 hours or autoclave for 20 minutes at 117° C. to 120° C.

Determination.

(*a*) Using an Absorptiometer.

FIG. 11.—TRANSFERRING THE DIGEST TO A 500 ML. VOLUMETRIC FLASK.
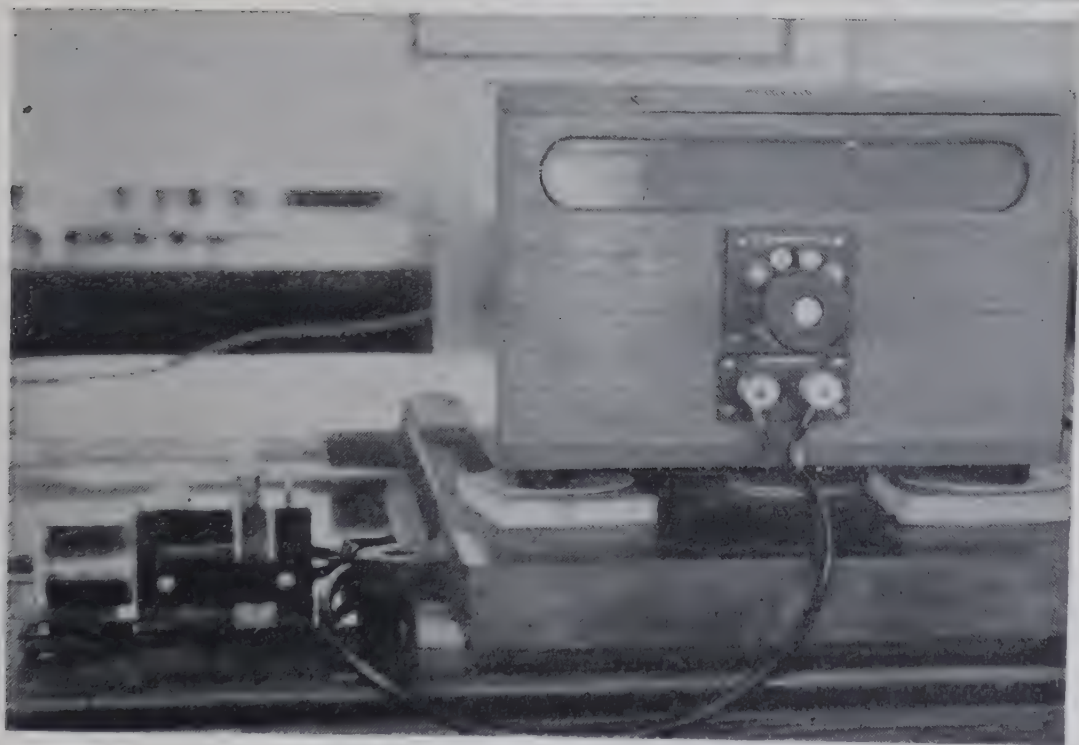
FIG. 12.—A PHOTOELECTRIC ABSORPTIOMETER.

Cool the hydrolysed solution and dilute to a volume of 500 ml. (Fig. 11).

Pipette an aliquot of 5 ml. into a clean dry 100 ml. volumetric flask and make up to 20 ml. with water. Add 20 ml. of 1 per cent picric acid and 2·6 ml. 2N sodium hydroxide.

Maintain at 20° C. ± 1° C. for 15 minutes. Dilute to 100 ml. with water. Filter (Whatman No. 12) and reject the first few millilitres until the solution is clear and bright.

Readings are made in the absorptiometer (Fig. 12) with an Ilford filter No. 604 and a 1 cm. cell; a reagent blank composed of all the reagents minus the creatinine is used. The colour can be read immediately and is stable for 30 minutes.

Prepare a standard curve covering a range of 0 to 1·0 mg. of creatinine. Into clean, dry, volumetric flasks, measure quantities of 2, 4, 6, 8, and 10 ml. of the standard creatinine zinc chloride solution. These flasks contain creatinine in amounts of 0·2 to 1·0 mg. Add distilled water to bring the volume of solution in each flask to 20 ml. To each flask add 20 ml. of 1 per cent picric acid solution and 2·5 ml. of 2N sodium hydroxide. Maintain at 20° C. ± 1° C. for 15 minutes and dilute each volume to 100 ml. Note—1 per cent picric acid solution should be standardised against N/10 sodium hydroxide, phenol red being used as indicator.

(b) Using the Duboscq Colorimeter (Fig. 13).

Cool the hydrolysed solution and dilute to a volume of 250 ml.

Measure two aliquots of 7 ml. and 10 ml. into clean dry 100 ml. volumetric flasks. Make up to 20 ml. with distilled water. Add 20 ml. of 1 per cent picric acid solution and 2·8 ml. 2N sodium hydroxide to the 7 ml. aliquot and 2·9 ml. 2N sodium hydroxide to the 10 ml. aliquot. Maintain at 20° C. ± 1° C. for 15 minutes. Dilute to 100 ml. with water. Filter (Whatman No. 12) and reject the first few millilitres until the solution is clear and bright.

Compare with a standard made up at the same time and under the same conditions from 20 ml. standard creatinine zinc chloride solution (equivalent to 2 mg. of creatinine). The colour is stable for 30 minutes.

These two aliquots will indicate the approximate percentage of total creatinine; an aliquot can then be calculated which, after development of colour and dilution, will compare closely with the standard.

7. INSOLUBLE AND COAGULABLE SOLIDS.—Weigh 10 gm. of the extract into a 250 ml. beaker and add 125 ml. of hot water. Bring to the boil, and add 0·5 ml. of glacial acetic acid. Continue boiling for a minute. Transfer the beaker to a steam bath until contents have flocculated.

Filter hot through a dried weighed Whatman No. 4 paper, allowing the filtrate to collect in a 250 ml. volumetric flask (this filtrate is required for both the gelatin and amino-acid nitrogen determinations) and well wash the coagulated material with hot water. Dry the filter paper overnight at 100° C. and reweigh. Additional weight equals insoluble and coagulable solids from 10 gm. of extract.

8. FAT.—Place the filter paper containing the coagulum from the previous determination in a Whatman extraction thimble, and extract in a Soxhlet apparatus (Fig. 14) for 3 hours, using petroleum ether of range 40° C. to 60° C. b. pt.

Dry 1 hour on the water bath followed by 1 hour in an electric oven at 100° C.

9. INSOLUBLE AND COAGULABLE NITROGEN.—Transfer the residue from the fat determination to a Kjeldahl flask and determine N. by method described under section 5.
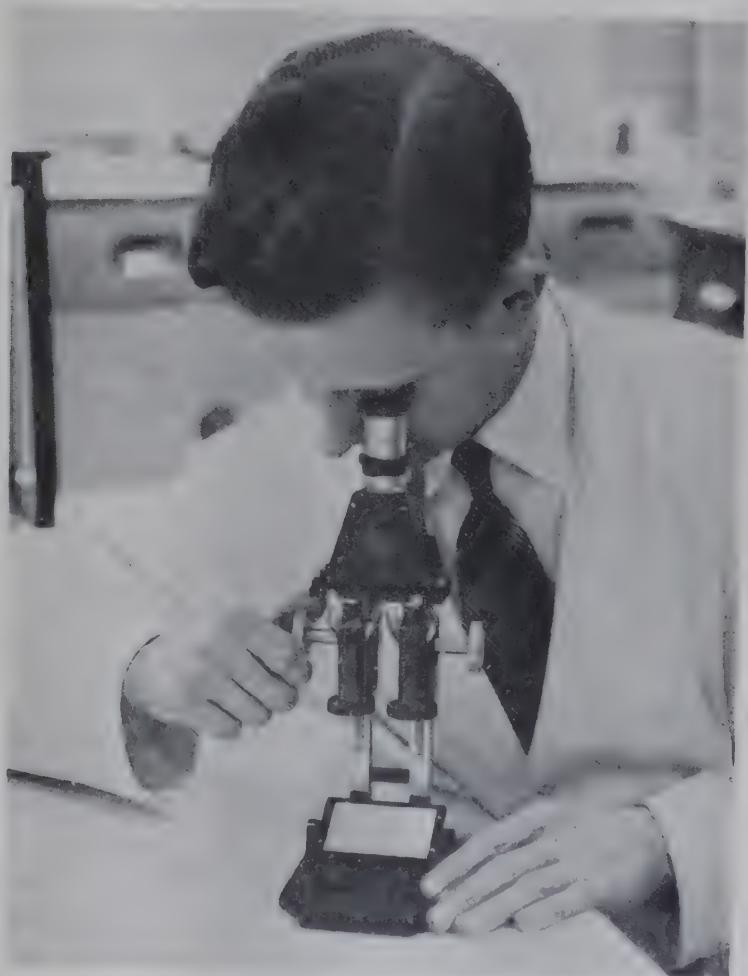
FIG. 13.—MATCHING THE COLOURS IN A DUBOSCQ COMPARATOR.

10. AMINO ACID NITROGEN.—Using N/2 sodium hydroxide solution, titrate potentiometrically (Fig. 15) to pH 8·4, 25 ml. of the filtrate from the coagulable solids determination.

Add 10 ml. of 40 per cent formaldehyde solution (previously neutralised potentiometrically to pH 8·4) and then titrate the mixture potentiometrically again to pH 8·4.

1 ml. N/2 NaOH ≡ 7 mg. amino acid N.

11. GELATIN.—Pipette 25 ml. of the filtrate from the insoluble and coagulable solids determination (Section 7) into a porcelain basin (capacity ca. 190 ml. Royal Worcester, Form 5. No. 4 is suitable). Add 0·25 ml. of 40 per cent formaldehyde solution and mix well.

Concentrate to a thick consistency, add 0·25 ml. of 40 per cent formaldehyde solution and stir thoroughly with a glass rod. Smear the mass over the inner surface of the basin to within 1 in. of the rim with the glass rod then bake hard on a vigorously boiling steam bath for 2 hours. Extract the contents of the dish twice with 100 ml. of 1 per cent formaldehyde solution at 40° C. allowing one hour for each extraction and maintaining the temperature at 40° C. during the extraction. Filter each washing through a Whatman No. 54 paper and during the final extraction break the complex up and loosen it from the dish. Transfer the complex to the filter paper and wash with 100 ml. 1 per cent formaldehyde solution at 40° C. Determine N. by the Kjeldahl method. The factor 5·55 is used to convert N. to gelatin.

12. CHEMICAL DETERMINATION OF NICOTINIC ACID.—REAGENTS.

Nicotinic acid primary standard—Dissolve 50 mg. of nicotinic acid in 50 per cent alcohol and make up to 200 ml. with the same solvent. This solution is stable for many months if stored in tightly-stoppered bottles in the refrigerator.

Nicotinic acid secondary standard —Prepare daily by diluting 10 ml. of primary standard to 250 ml. with water. It contains 10 μg. per ml.

Phosphate buffer solution—A 2 per cent solution of AnalaR potassium dihydrogen phosphate in distilled water. The pH is 4·6 and the solution is stable for long periods in a refrigerator.

Cyanogen bromide reagent—Make an approximately 4 per cent aqueous solution of cyanogen bromide by just decolorising ice-cold saturated bromine water with chilled 10 per cent sodium cyanide solution. The bromine water and sodium cyanide solution should be made up every few days and stored in a refrigerator and the cyanogen bromide prepared just before use. Care must be taken to avoid an excess of sodium cyanide in the final solution.

Procaine hydrochloride solution— Dissolve 3·5 gm. in 80 ml. of water, add 1·5 ml. of AnalaR concentrated hydrochloric acid
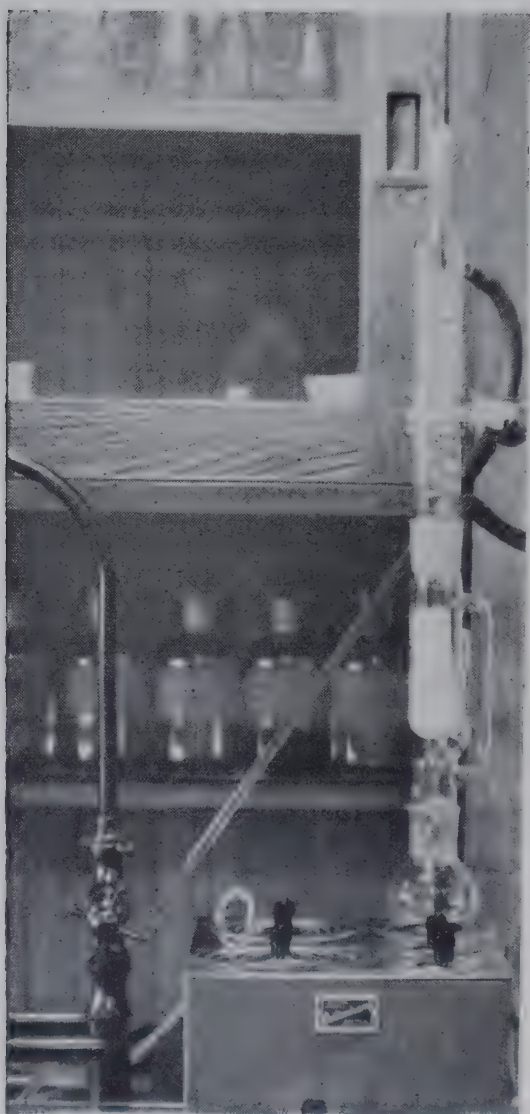
FIG. 14.—
A SOXHLET EXTRACTION APPARATUS.

and make up with water to 100 ml. The solution is stable in light and air, but is best made up daily.

METHOD.—Weigh out a quantity of the material containing between 1500 and 2500 μg. of nicotinic acid and digest it with 25 ml. of 4N caustic soda on the steam-bath for 30 to 40 minutes. Cool and adjust to pH 4·0 with concentrated hydrochloric acid, using bromophenol blue as external indicator; cool, make up with water to 100 ml. and filter through sintered glass (Fig. 16) to obtain a bright solution. The filtrate contains between 15 and 25 μg. of vitamin per ml. and, although coloured, is suitable for analysis without further treatment. One ml. aliquots of this test solution are taken for analysis.

The following mixtures are set up in 25 ml. volumetric flasks:

	T_1 ml.	T_2 ml.	S_1 ml.	S_2 ml.	S_3 ml.
Test solution . . .	1·0	1·0	—	—	—
Nicotinic acid soln. 10 μg./ml.	—	—	1·5	2·0	2·5
Water	1·5	1·5	1·0	0·5	—
Phosphate buffer solution pH 4·6	5·0	5·0	5·0	5·0	5·0

Heat the flasks in a water-bath to 60° C. and add to each 2 ml. of cyanogen bromide reagent, continue heating for 5 minutes, remove, cool to room temperature. Add 10 ml. procaine reagent to T_1 and T_2, make up to 25 ml. with water and measure the colour within 2 to 3 minutes in a photo-electric colorimeter using an Ilford Micro 1 blue filter, for under these conditions colour development is almost instantaneous. To flasks S_1, S_2 and S_3 add 10 ml. of procaine reagent followed by 1 ml. of the test extract, make up to 25 ml. with water and measure the colour intensity. Plot a graph of the galvonometer readings (i.e., $F_o \times 100$) of S_1, S_2 and S_3 against the nicotinic acid contents of the tubes, viz. 15, 20 and 25 μg. This should give a straight or very nearly straight line. From this the nicotinic acid contents of T_1 and T_2 may be read directly.

13. PHOSPHORUS (TOTAL).—REAGENTS.

(a) *Molybdate solution.*—Dissolve 100 gm. of molybdenum tri-oxide in a mixture of 144 ml. of ·880 ammonium hydroxide and 271 ml. of water. Cool and pour solution slowly and with constant stirring into a cool mixture of 489 ml. of concentrated nitric acid and 1148 ml. of water. Keep final mixture in a warm place for several days or until a portion heated to 40° C. deposits no yellow precipitate of ammonium phosphomolybdate. Decant solution from any sediment and preserve in glass stoppered vessels.

(b) *Ammonium Nitrate solution.*—Dissolve 100 gm. of phosphate-free ammonium nitrate in water and dilute to 1 litre.

(c) *Magnesia Mixture.*—Dissolve 11 gm. magnesium oxide in hydrochloric acid (1 + 4), avoiding an excess of the acid; add a little magnesium oxide in excess; boil for a few minutes to precipitate iron, aluminium and phosphate and filter.

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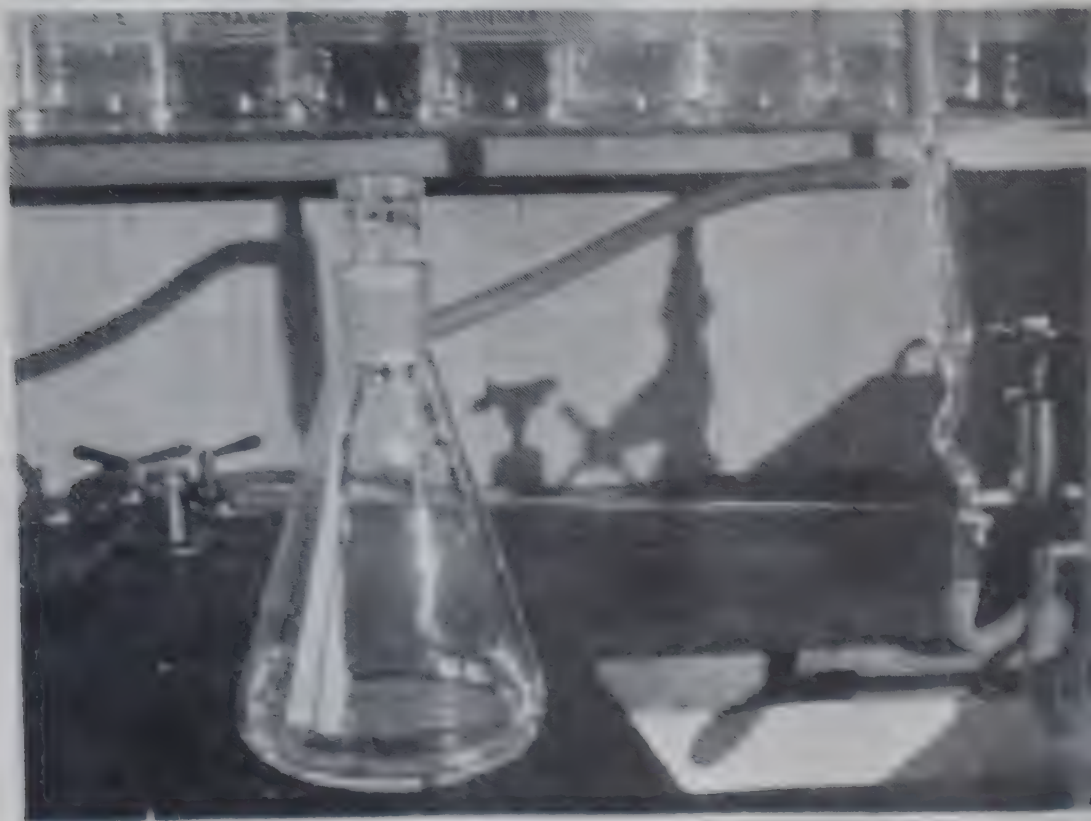
To filtrate add 140 gm. ammonium chloride and 199·5 ml. of ammonium hydroxide and dilute to 1 litre.

(d) *Ammonium Hydroxide solution for washing* (1 + 9).—Should contain not less than 2·5 per cent. of ammonia by weight.

(e) *Magnesium Nitrate solution.*—Dissolve 50 gm. of magnesium oxide in nitric acid (1 + 1), avoiding an excess of acid; add a little magnesium oxide in excess, boil, filter from excess magnesium oxide, iron oxide, etc., and dilute to 1 litre.

Method.—Dissolve 5 gm. of the extract in 12·5 ml. of the magnesium nitrate solution. Evaporate and ignite to ash. Dissolve the ash in hydrochloric acid. Dilute to 200 ml. Mix, and pour on to a dry Whatman 54 paper. Pipette 25 ml. of the filtrate into a 250 ml. beaker, add ammonium hydroxide in slight excess, and barely dissolve the precipitate formed with a few drops of nitric acid, stirring vigorously. Add 15 gm. of crystalline ammonium nitrate. To the hot solution add 70 ml. of the molybdate solution. Digest at ca. 65° C. for 1 hour. Filter (Whatman No. 7), and wash with the ammonium nitrate solution. Dissolve the precipitate on the filter with ammonium hydroxide (1 + 1) and hot water and wash into a beaker to a volume of not more than 100 ml. Neutralise with hydrochloric acid, using bromothymol blue as indicator; cool; and from a burette add slowly (ca. 1 drop per second), while stirring vigorously, 15 ml. of the magnesia mixture. After 15 minutes add 12 ml. of ammonium hydroxide. Let stand until supernatant liquid is clear (usually 2 hours), filter (Whatman No. 1), wash precipitate with ammonium hydroxide (1 + 9) until the washings are practically free from chlorides, dry, burn at a low heat, ignite to constant weight, preferably in an electric furnace, at 950° C. to 1000° C.; cool in a desiccator, and weigh as $Mg_2P_2O_7$. Report as percentage of P_2O_5. Conversion factor $Mg_2P_2O_7$ to P_2O_5 = 0·6379.

Meat Pastes

Introduction.—The essential ingredients of meat pastes (apart from meat) are:

1. Fat, which ensures the paste having good spreading properties.
2. Filler, i.e., farinaceous material to absorb excessive moisture and give a satisfactory texture.
3. Flavouring and seasoning.
4. Colouring. Usually this is Rhodamine B (the hydrochloride of diethyl-m-amino-phenol phthalein).

1. Sampling.—Turn complete contents of jar on to a plate, and mix thoroughly with a palette knife (Fig. 17). Replace immediately in original jar, and lose no time in weighing out quantities required for analyses. Care must be taken to prevent loss of water in sampling and subsequent handling of the sample.

2. Moisture.—Weigh into a tared nickel basin 5 gm. of the paste. Dry to constant weight at 100° C. in oven.

FIG. 17.—MIXING THE PASTE SAMPLE PRIOR TO ANALYSIS.

3. FAT.—Transfer the residue from the moisture determination to a Whatman extraction thimble, and extract with b. pt. 40 60° C. petrol for 3 hours. Dry 1 hour on the steam bath, followed by 1 hour in the 100° C. electric oven.

4. TOTAL NITROGEN AND PROTEIN.—The method described in the Meat Extract Section is used, except that 5 gm. of paste are taken.

5. ASH.—Again, the method described in the Meat Extract Section is used, taking 5 gm. of paste.

6. STARCH.—Digest 20 gm. of the sample with 300 ml. of 5 per cent alcoholic potash solution on a water-bath until the meat and fat have dissolved. Filter off the insoluble matter (through a Whatman 54 paper) and wash the filter with alcoholic potash. Wash the insoluble matter from the filter-paper into a beaker with 200 ml. of warm water. Add 40 ml. of N. aqueous potassium hydroxide solution and warm until the starch has dissolved. Cool the solution, transfer to a 250 ml. graduated flask and make up to volume with water. Filter a portion through a Whatman 54 paper.

Transfer 50 ml. of the solution to a beaker containing 300 ml. of 90 per cent by volume alcohol acidified with 0·5 ml. glacial acetic acid (i.e., sufficient to neutralise 8 ml. of N. potassium hydroxide). Stir well, and allow the starch to settle overnight. Filter off the precipitated starch through a Whatman 50 paper, wash it free from acid with 90 per cent alcohol, wash with ether to remove alcohol and dry at 100° C. After weighing, incinerate and deduct the ash from the weight of starch.

7. CALCULATIONS.

1. The sum of the percentages of water, fat, protein and ash deducted from 100 gives the percentage of dry carbohydrate and crude cellulose material.

2. From the total nitrogen deduct* an amount equal to 2 per cent of the dry carbohydrate plus crude cellulose for nitrogen associated with the carbohydrate and calculate the remainder to lean or defatted meat by means of the factors:

Beef and Mutton   .   .   .   . 100/3·4
Pork   .   .   .   .   . 100/3·6
Mixed Meat† .   .   .   . 100/3·5

3. The total meat present is the sum of the fat and the lean meat.

4. Calculate the approximate percentage of "cereal filler," containing 40 per cent of its weight of water, by multiplying the percentage of carbohydrate by two.

5. The difference between 100 and the sum of the percentages of "cereal filler," salt and total meat gives the additional water; that is water used in the preparation of the product other than natural moisture and water present in the filler.

   The determined starch should agree with the carbohydrate figure obtained as a difference figure by the Stubbs and More (Analyst 1919, 125) method to within ± 2 per cent.   If this is not so, the presence of substances such as milk powder or soya flour is indicated.

Corned Beef

1. SAMPLING.—After opening the can and expelling the meat, the debris adhering to the sides and ends of the can is scraped off and added to the bulk of the sample.   The whole is then finely minced three times.

2. MOISTURE.—Use the method described under Meat Pastes.

3. ASH.—Use the method described under Meat Pastes.

4. CHLORIDE AS SODIUM CHLORIDE.—Use the method described under Meat Pastes.

5. NITROGEN.—Use the method described under Meat Pastes.

6. STARCH.—(Required only when sample is corned beef with cereal.)

7. NITRATE.—REAGENTS.

Alumina Cream—Add a slight excess of strong ammonia solution to a saturated aqueous solution of aluminium potassium sulphate A.R., and then cautiously add more alum solution until the mixture is just acid to litmus.

* If the filler consists of potato starch no correction for nitrogen is made.

† That is, pork and beef, and meat unspecified, e.g., meat roll and luncheon meat.

Lead Acetate—(*Basic*) Solution 25 per cent w/w.

Sodium Hydroxide—Solution 2N.

Sulphuric Acid—85 per cent w/w (nitrogen free).

2:4 – Xylen-1-ol—Solution 1 per cent w/v in glacial acetic acid.

Silver Sulphate—(Nitrogen free) A.R.

METHOD.—Transfer 1 gram of the minced sample to a 100 ml. volumetric flask containing 20 ml. of water, immerse the flask in a boiling water bath for 15 minutes and periodically agitate the contents. Allow the mixture to cool, render just acid with dilute sulphuric acid using bromocresol green as indicator and oxidise any nitrates which may be present by adding 0·2N potassium permanganate drop by drop until a faint pink colour persists for 1 minute. Add successively, with shaking after each addition, sufficient saturated aqueous solution of silver sulphate AnalaR to precipitate any chloride present, 5 ml. of 25 per cent w/v solution of basic lead acetate and 5 ml. of alumina cream. Dilute with water to 100 ml., again shake and filter. Mix an appropriate proportion of the filtrate not exceeding 20 ml. (usually from 1 ml. to 5 ml. will be suitable) with three times its volume of 85 per cent w/w sulphuric acid, adjust the temperature of the mixture to 35° C., add 1 ml. of the xylenol solution, maintain at 35° C. for half an hour, then dilute with 100 ml. of distilled water and transfer to an ordinary distillation apparatus (Fig. 18). Distil the mixture and
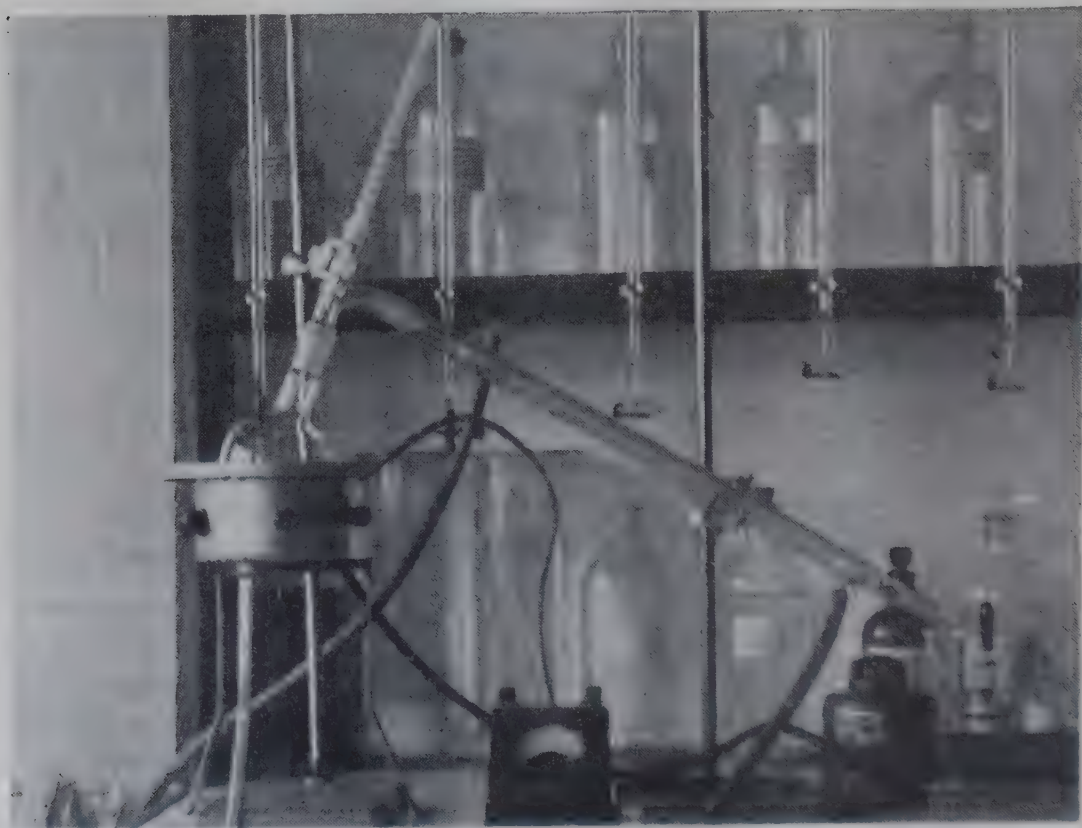
FIG. 18.—APPARATUS FOR THE DETERMINATION OF NITRATE BY THE METAXYLENOL METHOD.

collect 50 ml. in a receiver containing 10 ml. of 2N sodium hydroxide and dilute the distillate to an appropriate volume. Transfer 50 ml. to a Nessleriser glass, adjust the temperature of the liquid to 20° C. and place the Nessleriser glass in the right-hand compartment of the Nessleriser. Fill another glass to the 50 ml. mark with water and place in the left-hand compartment of the instrument. Stand the Nessleriser before a uniform source of white light—a north window wherever possible is the best—and compare the colour produced in the test solution with the colours in the standard disc, rotating the disc until a match is obtained.

The markings on the disc represent the actual amounts of nitrate nitrogen (N) producing the colour in the test. Thus, if a colour equivalent to 8 is produced, the amount of nitrogen present as nitrate in the quantity of solution taken for the test is 0·008 milligram.

It is important that the temperature of distillates should be adjusted to 20° C. which is the temperature adopted for the standardisation of the nitrate disc, since the colour intensity of the nitro-compound in alkaline solution increases by 0·62 per cent per 1° C.

<div align="right">C. D. E.
J. H. S.</div>

INDEX

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